

<b>ENGINEERING CHANGE NOTICE</b>	1. ECN <b>643868</b> <hr/> Proj. ECN
Page 1 of <u>2</u> (5 KB 1/26/99)	

2. ECN Category (mark one)  Supplemental <input type="checkbox"/> Direct Revision <input checked="" type="checkbox"/> Change ECN <input type="checkbox"/> Temporary <input type="checkbox"/> Standby <input type="checkbox"/> Supersedure <input type="checkbox"/> Cancel/Void <input type="checkbox"/>	3. Originator's Name, Organization, MSIN, and Telephone No. Jim G. Field, Data Assessment and Interpretation, R2-12, 376-3753	4. USQ Required? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	5. Date 01/19/99
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13a. Description of Change The document has been totally revised to include the results of recent sampling to address technical issues associated with the waste, and to update the best basis standard inventory.	13b. Design Baseline Document? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
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14a. Justification (mark one)					
Criteria Change <input checked="" type="checkbox"/>	Design Improvement <input type="checkbox"/>	Environmental <input type="checkbox"/>	Facility Deactivation <input type="checkbox"/>		
As-Found <input type="checkbox"/>	Facilitate Const <input type="checkbox"/>	Const. Error/Omission <input type="checkbox"/>	Design Error/Omission <input type="checkbox"/>		

14b. Justification Details Changes required to incorporate new sampling data.
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## ENGINEERING CHANGE NOTICE

Page 2 of 2

1. ECN (use no. from pg. 1)

ECN-643868

## 16. Design Verification Required

☐ Yes  
☒ No

## 17. Cost Impact

## ENGINEERING

## CONSTRUCTION

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Functional Design Criteria	<input type="checkbox"/>	Stress/Design Report	<input type="checkbox"/>	Health Physics Procedure	<input type="checkbox"/>
Operating Specification	<input type="checkbox"/>	Interface Control Drawing	<input type="checkbox"/>	Spares Multiple Unit Listing	<input type="checkbox"/>
Criticality Specification	<input type="checkbox"/>	Calibration Procedure	<input type="checkbox"/>	Test Procedures/Specification	<input type="checkbox"/>
Conceptual Design Report	<input type="checkbox"/>	Installation Procedure	<input type="checkbox"/>	Component Index	<input type="checkbox"/>
Equipment Spec.	<input type="checkbox"/>	Maintenance Procedure	<input type="checkbox"/>	ASME Coded Item	<input type="checkbox"/>
Const. Spec.	<input type="checkbox"/>	Engineering Procedure	<input type="checkbox"/>	Human Factor Consideration	<input type="checkbox"/>
Procurement Spec.	<input type="checkbox"/>	Operating Instruction	<input type="checkbox"/>	Computer Software	<input type="checkbox"/>
Vendor Information	<input type="checkbox"/>	Operating Procedure	<input type="checkbox"/>	Electric Circuit Schedule	<input type="checkbox"/>
OM Manual	<input type="checkbox"/>	Operational Safety Requirement	<input type="checkbox"/>	ICRS Procedure	<input type="checkbox"/>
FSAR/SAR	<input type="checkbox"/>	IEFD Drawing	<input type="checkbox"/>	Process Control Manual/Plan	<input type="checkbox"/>
Safety Equipment List	<input type="checkbox"/>	Cell Arrangement Drawing	<input type="checkbox"/>	Process Flow Chart	<input type="checkbox"/>
Radiation Work Permit	<input type="checkbox"/>	Essential Material Specification	<input type="checkbox"/>	Purchase Requisition	<input type="checkbox"/>
Environmental Impact Statement	<input type="checkbox"/>	Fac. Proc. Samp. Schedule	<input type="checkbox"/>	Tickler File	<input type="checkbox"/>
Environmental Report	<input type="checkbox"/>	Inspection Plan	<input type="checkbox"/>		<input type="checkbox"/>
Environmental Permit	<input type="checkbox"/>	Inventory Adjustment Request	<input type="checkbox"/>		<input type="checkbox"/>

## 20. Other Affected Documents: (NOTE: Documents listed below will not be revised by this ECN.) Signatures below indicate that the signing organization has been notified of other affected documents listed below.

Document Number/Revision

Document Number/Revision

Document Number Revision

N/A

## 21. Approvals

Signature	Date	Signature	Date
Design Authority		Design Agent	
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## DEPARTMENT OF ENERGY

Signature or a Control Number that tracks the Approval Signature

## ADDITIONAL

# Tank Characterization Report for Single-Shell Tank 241-AX-104

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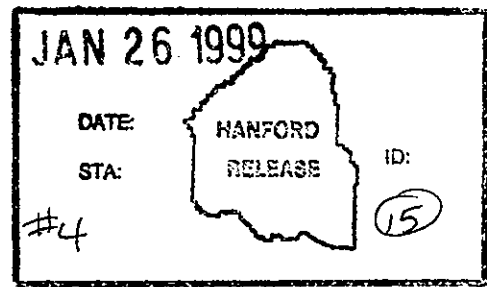
Abstract: This document summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in Tank 241-AX-104. This report supports the requirements of the Tri-Party Agreement Milestone M-44-15C.

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*Chris Braden*  
Release Approval

1/26/99  
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# Tank Characterization Report for Single-Shell Tank 241-AX-104

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## CONTENTS

1.0 INTRODUCTION .....	1-1
1.1 SCOPE.....	1-1
1.2 TANK BACKGROUND.....	1-1
2.0 RESPONSE TO TECHNICAL ISSUES .....	2-1
2.1 TANK 241-AX-104 WASTE CHARACTERIZATION DATA QUALITY OBJECTIVE.....	2-1
2.2 SAFETY SCREENING.....	2-2
2.2.1 Exothermic Conditions (Energetics) .....	2-2
2.2.2 Flammable Gas.....	2-3
2.2.3 Criticality.....	2-3
2.3 ORGANIC COMPLEXANTS .....	2-4
2.4 ORGANIC SOLVENTS SAFETY SCREENING .....	2-4
2.5 OTHER TECHNICAL ISSUES.....	2-4
2.5.1 Hazardous Vapor Screening.....	2-4
2.5.2 Tank Waste Heat Load.....	2-5
2.6 SUMMARY .....	2-6
3.0 BEST-BASIS STANDARD INVENTORY ESTIMATE .....	3-1
4.0 RECOMMENDATIONS.....	4-1
5.0 REFERENCES .....	5-1
APPENDIX A: HISTORICAL TANK INFORMATION .....	A-1
A1.0 CURRENT TANK STATUS.....	A-3
A2.0 TANK DESIGN AND BACKGROUND .....	A-4
A3.0 PROCESS KNOWLEDGE.....	A-9
A3.1 WASTE TRANSFER HISTORY.....	A-9
A3.2 HISTORICAL ESTIMATION OF TANK CONTENTS .....	A-10
A4.0 SURVEILLANCE DATA .....	A-15
A4.1 SURFACE-LEVEL READINGS.....	A-15
A4.2 INTERNAL TANK TEMPERATURES.....	A-16
A4.3 TANK 241-AX-104 PHOTOGRAPHS.....	A-16
A5.0 APPENDIX A REFERENCES.....	A-21
APPENDIX B: SAMPLING OF TANK 241-AX-104.....	B-1
B1.0 TANK SAMPLING OVERVIEW .....	B-3
B2.0 SAMPLING EVENTS .....	B-4

**CONTENTS (Continued)**

B2.1 DESCRIPTION OF 1997 AUGER SAMPLING EVENT .....	B-5
B2.1.1 Sample Handling .....	B-6
B2.1.2 Sample Analysis .....	B-9
B2.1.3 Analytical Results .....	B-14
B2.2 VAPOR PHASE MEASUREMENTS .....	B-22
B2.3 DESCRIPTION OF 1997 VAPOR SAMPLING EVENT .....	B-23
B2.4 DESCRIPTION OF HISTORICAL SAMPLING EVENT .....	B-26
B2.4.1 June 1978 Data Set .....	B-26
B2.4.2 September 1977 Sludge Sampling .....	B-27
B2.5 DATA TABLES FOR THE NOVEMBER 1997 AUGER SAMPLES .....	B-30
 B3.0 ASSESSMENT OF CHARACTERIZATION RESULTS .....	 B-49
B3.1 FIELD OBSERVATIONS .....	B-49
B3.2 QUALITY CONTROL ASSESSMENT .....	B-52
B3.3 DATA OCNSISTENCY CHECKS .....	B-55
B3.3.1 Comparison of Results from Different Analytical Methods .....	B-55
B3.3.2 Mass and Charge Balances .....	B-55
B3.4 MEAN CONCENTRATIONS AND CONFIDENCE INTERVALS .....	B-58
 B4.0 APPENDIX B REFERENCES .....	 B-62
 APPENDIX C: STATISTICAL ANALYSIS FOR ISSUE RESOLUTION .....	 C-1
C1.0 STATISTICS FOR THE SAFETY SCREENING DATA QUALITY OBJECTIVE .....	C-3
C2.0 APPENDIX C REFERENCES .....	C-5
 APPENDIX D: EVALUATION TO ESTABLISH BEST-BASIS INVENTORY FOR SINGLE-SHELL TANK 241-AX-104 .....	 D-1
D1.0 CHEMICAL INFORMATION SOURCES .....	D-3
D2.0 COMPARISON OF COMPONENT INVENTORY VALUES .....	D-4
D3.0 COMPONENT INVENTORY EVALUATION .....	D-7
D3.1 WASTE TYPES .....	D-8
D3.2 SUMMARY .....	D-8
D4.0 DEFINE THE BEST-BASIS AND ESTABLISH COMPONENT INVENTORIES .....	D-9
D5.0 APPENDIX D REFERENCES .....	D-13
 APPENDIX E: BIBLIOGRAPHY FOR TANK 241-AX-104 .....	 E-1

## LIST OF FIGURES

A2-1 Riser Configuration for Tank 241-AX-104. ....	A-7
A2-2 Tank 241-AX-104 Cross Section and Schematic. ....	A-8
A3-1 Tank Layer Model. ....	A-11
A4-1 Tank 241-AX-104 Level History Through 1995.....	A-18
A4-2 Tank 241-AX-104 Level History Since January 1996. ....	A-19
A4-3 Tank 241-AX-104 Temperature Profile. ....	A-20

## LIST OF TABLES

1-1 Summary of Recent Sampling.....	1-2
1-2 Description of Tank 241-AX-104.....	1-3
2-1 Projected Heat Load. ....	2-5
2-2 Summary of Technical Issues.....	2-6
3-1 Best-Basis Inventory Estimates for Nonradioactive Components in Tank 241-AX-104.....	3-2
3-2 Best-Basis Inventory Estimates for Radioactive Components in Tank 241-AX-104, Decayed to January 1, 1994 .....	3-3
4-1 Acceptance of Tank 241-AX-104 Sampling and Analysis. ....	4-1
4-2 Acceptance of Evaluation of Characterization Data and Information for Tank 241-AX-104.....	4-2
A1-1 Tank Contents Status Summary.....	A-4
A2-1 Riser Descriptions for Tank 241-AX-104 .....	A-5
A3-1 Tank 241-AX-104 Major Transfers.....	A-10
A3-2 Hanford Defined Waste Model Tank Inventory Estimate: Analytes.....	A-12



**LIST OF TABLES (Continued)**

A3-3 Hanford Defined Waste Model Tank Inventory Estimate: Radionuclides.....	A-13
B2-1 Integrated Data Quality Objective Requirements for Tank 241-AX-104.....	B-4
B2-2 Extrusion Information and Sample Descriptions.....	B-6
B2-3 Analytical Procedures .....	B-11
B2-4 Sample Analysis Summary for Whole Samples .....	B-12
B2-5 Sample Analysis Summary for Composite and Leach Test Samples.....	B-13
B2-6 Analytical Tables .....	B-14
B2-7 Results of ICP/MS Screen .....	B-20
B2-8 Composite and Leach Test Results for the Required Analytes.....	B-21
B2-9 Results of Headspace Measurements of Tank 241-AX-104.....	B-22
B2-10 Headspace Vapor Analytical Results for Tank 241-AX-104 .....	B-24
B2-11 1978 Data Set.....	B-26
B2-12 Analytical Results from 1977 Sludge Sampling.....	B-28
B2-13 Physical Property Analytical Results for the 1977 Sample .....	B-29
B2-14 Tank 241-AX-104 Analytical Results: Aluminum (ICP).....	B-30
B2-15 Tank 241-AX-104 Analytical Results: Antimony (ICP).....	B-31
B2-16 Tank 241-AX-104 Analytical Results: Arsenic (ICP).....	B-31
B2-17 Tank 241-AX-104 Analytical Results: Barium (ICP). .....	B-31
B2-18 Tank 241-AX-104 Analytical Results: Beryllium (ICP). .....	B-32
B2-19 Tank 241-AX-104 Analytical Results: Bismuth (ICP).....	B-32
B2-20 Tank 241-AX-104 Analytical Results: Boron (ICP). .....	B-32

**LIST OF TABLES (Continued)**

B2-21	Tank 241-AX-104 Analytical Results: Cadmium (ICP).....	B-33
B2-22	Tank 241-AX-104 Analytical Results: Calcium (ICP).....	B-33
B2-23	Tank 241-AX-104 Analytical Results: Cerium (ICP). ....	B-33
B2-24	Tank 241-AX-104 Analytical Results: Chromium (ICP) .....	B-34
B2-25	Tank 241-AX-104 Analytical Results: Cobalt (ICP).....	B-34
B2-26	Tank 241-AX-104 Analytical Results: Copper (ICP).....	B-34
B2-27	Tank 241-AX-104 Analytical Results: Iron (ICP).....	B-35
B2-28	Tank 241-AX-104 Analytical Results: Lanthanum (ICP). ....	B-35
B2-29	Tank 241-AX-104 Analytical Results: Lead (ICP). ....	B-35
B2-30	Tank 241-AX-104 Analytical Results: Lithium (ICP).....	B-36
B2-31	Tank 241-AX-104 Analytical Results: Magnesium (ICP).....	B-36
B2-32	Tank 241-AX-104 Analytical Results: Manganese (ICP). ....	B-36
B2-33	Tank 241-AX-104 Analytical Results: Molybdenum (ICP).....	B-37
B2-34	Tank 241-AX-104 Analytical Results: Neodymium (ICP). ....	B-37
B2-35	Tank 241-AX-104 Analytical Results: Nickel (ICP).....	B-37
B2-36	Tank 241-AX-104 Analytical Results: Phosphorus (ICP).....	B-38
B2-37	Tank 241-AX-104 Analytical Results: Potassium (ICP).....	B-38
B2-38	Tank 241-AX-104 Analytical Results: Samarium (ICP). ....	B-38
B2-39	Tank 241-AX-104 Analytical Results: Selenium (ICP). ....	B-39
B2-40	Tank 241-AX-104 Analytical Results: Silicon (ICP).....	B-39
B2-41	Tank 241-AX-104 Analytical Results: Silver (ICP).....	B-39
B2-42	Tank 241-AX-104 Analytical Results: Sodium (ICP).....	B-40

**LIST OF TABLES (Continued)**

B2-43 Tank 241-AX-104 Analytical Results: Strontium (ICP). .....	B-40
B2-44 Tank 241-AX-104 Analytical Results: Sulfur (ICP). .....	B-40
B2-45 Tank 241-AX-104 Analytical Results: Thallium (ICP). .....	B-41
B2-46 Tank 241-AX-104 Analytical Results: Titanium (ICP). .....	B-41
B2-47 Tank 241-AX-104 Analytical Results: Total Uranium (ICP). .....	B-41
B2-48 Tank 241-AX-104 Analytical Results: Vanadium (ICP). .....	B-42
B2-49 Tank 241-AX-104 Analytical Results: Zinc (ICP). .....	B-42
B2-50 Tank 241-AX-104 Analytical Results: Zirconium (ICP). .....	B-42
B2-51 Tank 241-AX-104 Analytical Results: Bromide (IC). .....	B-43
B2-52 Tank 241-AX-104 Analytical Results: Chloride (IC). .....	B-43
B2-53 Tank 241-AX-104 Analytical Results: Fluoride (IC). .....	B-43
B2-54 Tank 241-AX-104 Analytical Results: Nitrate (IC). .....	B-44
B2-55 Tank 241-AX-104 Analytical Results: Nitrite (IC). .....	B-44
B2-56 Tank 241-AX-104 Analytical Results: Oxalate (IC). .....	B-44
B2-57 Tank 241-AX-104 Analytical Results: Phosphate (IC). .....	B-45
B2-58 Tank 241-AX-104 Analytical Results: Sulfate (IC). .....	B-45
B2-59 Tank 241-AX-104 Analytical Results: Americium-241 (AEA). .....	B-45
B2-60 Tank 241-AX-104 Analytical Results: Cesium-137 (GEA). .....	B-46
B2-61 Tank 241-AX-104 Analytical Results: Cobalt-60 (GEA). .....	B-46
B2-62 Tank 241-AX-104 Analytical Results: Plutonium-239/240 (AEA). .....	B-46
B2-63 Tank 241-AX-104 Analytical Results: Selenium-79 (Liquid Scintillation) .....	B-47
B2-64 Tank 241-AX-104 Analytical Results: Strontium-89/90 (Beta Counting). .....	B-47

**LIST OF TABLES (Continued)**

B2-65	Tank 241-AX-104 Analytical Results: Technetium-99 (Liquid Scintillation).....	B-48
B2-66	Tank 241-AX-104 Analytical Results: Total Alpha (Alpha Counting).....	B-48
B2-67	Tank 241-AX-104 Analytical Results: Percent Water.....	B-48
B2-68	Tank 241-AX-104 Analytical Results: Total Organic Carbon (Furnace Oxidation).....	B-49
B3-1	Comparison Of Data For Key Analytes.....	B-50
B3-2	Cation Mass And Charge Data for Solids.....	B-57
B3-3	Anion Mass And Charge Data for Solids .....	B-58
B3-4	Mass And Charge Balance Totals.....	B-58
B3-5	Tank 241-AX-104 95 Percent Two-Sided Confidence Interval for The Mean Concentration For Solid Sample Data .....	B-60
C1-1	95 Percent Upper Confidence Limits for Total Alpha Activity and <sup>239/240</sup> Pu.....	C-4
C1-2	95 Percent Upper Confidence Limits for Total Organic Carbon .....	C-4
D2-1	Inventory Estimates for Selected Nonradioactive Components in Tank 241-AX-104.....	D-5
D2-2	Inventory Estimates for Radioactive Components in Tank 241-AX-104.....	D-6
D4-1	Best-Basis Inventory Estimates for Nonradioactive Components in Tank 241-AX-104.....	D-10
D4-2	Best-Basis Inventory Estimates for Radioactive Components in Tank 241-AX-104, Decayed to January 1, 1994 .....	D-11

## LIST OF TERMS

AEA	alpha energy analysis
AES	atomic emission spectroscopy
ALARA	as low as reasonably achievable
AMU	atomic mass units
ANOVA	analysis of variance
B	acid waste from PUREX, processed through B Plant
Btu/hr	British thermal units per hour
Ci	curie
Ci/L	curies per liter
CI	confidence interval
cm	centimeter
df	degrees of freedom
DQO	data quality objective
DSC	differential scanning calorimetry
ft	feet
g	gram
GEA	gamma energy analysis
g/L	grams per liter
g/mL	grams per milliliter
HDW	Hanford defined waste
HTI	Hanford Tank Initiative
IC	ion chromatography
ICP	inductively coupled plasma spectroscopy
IWW	inorganic waste wash
in.	inch
J/g	joules per gram
kg	kilogram
kgal	kilogallon
kg/L	kilograms per liter
kL	kiloliter
kW	kilowatt
LA/MS	laser ablation/mass spectrometer
LDUA	light duty utility arm
LEL	lower explosive limit
LL	lower limit
m	meter
m <sup>2</sup>	square meters
M	moles per liter
mg/m <sup>3</sup>	milligrams per cubic meter
mL	milliliter
mm	millimeter
MS	mass spectrometry

**LIST OF TERMS (Continued)**

n/a	not applicable
n/d	not detected
n/r	not reported
OWW <sup>3</sup>	PUREX organic wash waste generated from 1968 to 1972
P2	PUREX high-level waste
PL	PUREX low-level waste
ppb	parts per billion
ppm	parts per million
ppbv	parts per billion by volume
ppmv	parts per million by volume
PHMC	Project Hanford Management Contractor
PUREX	plutonium-uranium extraction (facility)
QC	quality control
REML	restricted maximum likelihood method
RPD	relative percent difference
rpm	revolutions per minute
SAP	sampling and analysis plan
SMM	supernatant mixing model
TCR	tank characterization report
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TLM	tank layer model
TOC	total organic carbon
TWRS	Tank Waste Remediation System
UL	upper limit
vol%	volume percent
W	watt
W/Ci	watts per curie
W/L	watts per liter
WSTRS	Waste Status and Transaction Record Summary
wt%	weight percent
%	percent
°C	degrees Celsius
°F	degrees Fahrenheit
$\bar{\mu}$	mean
$\mu\text{Ci/g}$	microcuries per gram
$\mu\text{Ci/mL}$	microcuries per milliliter
$\mu\text{eq/g}$	microequivalents per gram
$\mu\text{g}$	microgram
$\mu\text{g C/g}$	micrograms of carbon per gram
$\mu\text{g/g}$	micrograms per gram
$\mu\text{g/mL}$	micrograms per milliliter

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## 1.0 INTRODUCTION

A major function of the Tank Waste Remediation System (TWRS) is to characterize waste in support of waste management and disposal activities at the Hanford Site. Analytical data from sampling and analysis and other available information about a tank are compiled and maintained in a tank characterization report (TCR). This report and its appendices serve as the TCR for single-shell tank 241-AX-104.

The objectives of this report are 1) to use characterization data in response to technical issues associated with tank 241-AX-104 waste, and 2) to provide a standard characterization of this waste in terms of a best-basis inventory estimate. Section 2.0 summarizes the response to technical issues, Section 3.0 shows the best-basis inventory estimate, and Section 4.0 makes recommendations about the safety status of the tank and additional sampling needs. The appendices contain supporting data and information. This report supports the requirements of the *Hanford Federal Facility Agreement and Consent Order* (Ecology et al. 1997) Milestone M-44-15c, change request M-44-97-03, to "issue characterization deliverables consistent with the Waste Information Requirements Document developed for FY 1999" (Adams et al. 1998b).

### 1.1 SCOPE

The characterization information in this report originated from sample analyses and known historical sources. Samples were obtained and assessed to fulfill requirements for tank-specific issues discussed in Section 2.0 of this report. Other information was used to support conclusions derived from these results. Appendix A contains historical information for tank 241-AX-104, including surveillance information, records pertaining to waste transfers and tank operations, and expected tank contents derived from a process knowledge model. Appendix B summarizes recent sampling events (see Table 1-1), sample data obtained before 1989, and sampling results. Appendix C provides the statistical analysis and numerical manipulation of data used in issue resolution. Appendix D contains the evaluation to establish the best basis for the inventory estimate. Appendix E is a bibliography that resulted from an in-depth literature search of all known information sources applicable to tank 241-AX-104 and its respective waste types.

### 1.2 TANK BACKGROUND

Tank 241-AX-104 is located in the 200 East Area AX Tank Farm on the Hanford Site. The AX Tank Farm contains the final generation of single-shell tanks built at the Hanford Site. These tanks were designed to contain self-boiling wastes, primarily from the plutonium-uranium extraction (PUREX) facility and the B Plant. Tank 241-AX-104 went into service in 1965, receiving water from miscellaneous sources (Agnew et al. 1997b). Waste was first received in the fourth quarter of 1965 from tank 241-A-102. Transfers between tanks 241-A-102 and 241-AX-104 continued throughout 241-AX-104's active service life. From 1966 through 1969, tank 241-AX-104 received several waste types from PUREX. A majority of this PUREX waste was high-level waste containing substantial amounts of fission products. Because of the high concentration of heat-generating cesium and strontium isotopes present, this waste self-boiled.



Table 1-1. Summary of Recent Sampling.

Sample/Date	Phase	Location	Segmentation	Recovery
Auger samples (11/13/97- 11/21/97) <sup>1</sup>	Solid	Risers 3A and 9G	None	Recovered between 36.9 and 96.8 g per auger sample
Combustible gas measurement (11/14/97)	Gas	Tank headspace, Riser 3A, 6.1 m (20 ft) below top of riser	n/a	n/a
Vapor samples (1/23/97)	Gas	Tank headspace, riser 3A, 11.6 m (38 ft) below top of riser adapter flange	n/a	n/a

## Notes:

n/a = not applicable

<sup>1</sup>Dates are in mm/dd/yy format.

From 1970 until the end of the tank's active service life in 1977, transfers of supernatant waste were made both to and from a variety of other single-shell tanks (Agnew et al. 1997b). A sluicing campaign was initiated in 1977 to remove the cesium and strontium isotopes, and most of the tank solids were removed at this time. A second sluicing campaign in 1978 further reduced the solids volume. Interim stabilization (1981) and intrusion prevention (1982) followed the sluicing campaigns (Brevick et al. 1997).

In 1977, tank 241-AX-104 was declared an assumed leaker because of increasing levels of radioactivity in dry wells surrounding the tank. The source of the leak was attributed to the tank's vapor line and vessel vent header (Brevick et al. 1997).

Table 1-2 is an overall description of tank 241-AX-104. The tank has a maximum storage capacity of 3,785 kL (1,000 kgal), and presently contains an estimated 28.4 kL (7.5 kgal) of noncomplexed waste (see Table 1-2). The tank is not on the Watch List (Public Law 101-510).

Table 1-2. Description of Tank 241-AX-104.

<b>TANK DESCRIPTION</b>	
Type	Single-shell
Constructed	1963-1964
In service	1965
Diameter	22.9 m (75.0 ft)
Operating depth	9.2 m (30.3 ft)
Capacity	3,785 kL (1,000 kgal)
Bottom shape	Flat
Ventilation	Passive
<b>TANK STATUS (as of 11/11/98)</b>	
Waste classification	Noncomplexed
Total waste volume <sup>1</sup>	28.4 kL (7.5 kgal)
Supernatant volume	0 kL (0 kgal)
Saltcake volume	0 kL (0 kgal)
Sludge volume	28.4 kL (7.5 kgal)
Drainable interstitial liquid volume	0 kL (0 kgal)
Waste surface level <sup>2</sup> (11/11/98) <sup>3</sup>	3.63 cm (1.43 in.)
Temperature (7/1/97 to 7/1/98) <sup>4</sup>	29.2 °C (84.5 °F) to 33.1 °C (91.6 °F)
Integrity	Assumed leaker
Watch List	None
Flammable Gas Facility Group	3
<b>SAMPLING DATE</b>	
Auger samples	November 1997
Vapor samples	January 1997
<b>SERVICE STATUS</b>	
Declared inactive	1978
Interim stabilization	1981
Intrusion prevention	1982

## Notes:

<sup>1</sup>Reich (1997) estimates the waste volume to be between 18.9 kL (5 kgal) and 28.4 kL (7.5 kgal). A best-basis inventory of 28.4 kL (7.5 kgal) is assumed for this report. This differs from the Hanlon (1998) estimate of 26.5 kL (7 kgal).

<sup>2</sup>Auto ENRAF installed in October 1996. Measurements are not consistent with tank volume estimates (see Section A4.1).

<sup>3</sup>Dates are in mm/dd/yy format.

<sup>4</sup>July 1, 1998, is the date of the most recent temperature measurement.

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## 2.0 RESPONSE TO TECHNICAL ISSUES

The following technical issue has been identified for tank 241-AX-104 (Brown et al. 1998).

- **Tank 241-AX-104:** What is the inventory and leachability of the waste in tank 241-AX-104 (Banning 1998)?

Additional technical issues required by Brown et al. (1997) and addressed by sampling events include:

- **Safety screening:** Does the waste pose or contribute to any recognized potential safety problems?
- **Organic complexants:** Does the possibility exist for a point source ignition in the waste followed by a propagation of the reaction in the solid/liquid phase of the waste?
- **Organic solvents:** Does an organic solvent pool exist that may cause a fire or ignition of organic solvents in entrained waste solids?

Data from the analysis of auger samples, tank headspace measurements, and tank vapor samples, along with available historical information, provided the means to respond to the technical issues. The following sections present the response. See Appendix B for sample and analysis data for tank 241-AX-104.

As described in Section B3.1, significant uncertainties exist regarding the representativeness of the riser 3A auger samples to the majority of the tank waste. To provide a radiologically conservative waste inventory, no means or confidence intervals were calculated using data from the riser 3A auger samples. However, because the riser 3A auger results do provide composition data for the waste under that particular riser, the results have been used in the safety screening assessment. Because analytical results from both risers 3A and 9G are used in the assessment, the safety screening data quality objective (DQO) requirement of two vertical waste profiles is considered to have been met.

### 2.1 TANK 241-AX-104 WASTE CHARACTERIZATION DATA QUALITY OBJECTIVE

Tank 241-AX-104 was selected as the preferred tank at which residual waste characterization could be conducted to support the Hanford Tanks Initiative (HTI) Project. The primary objective of the HTI Project is to provide a technical basis for the design and regulatory decisions for the waste retrieval and closure of high-level waste tanks at the Hanford Site. To meet the needs of the HTI Project, determination of the tank 241-AX-104 waste inventory was required, as well as a waste leachability study. *Tank 241-AX-104 Waste Characterization Data Quality Objective* (Banning 1998), referred to as the HTI DQO, was prepared to define the sampling and analytical requirements needed to resolve these issues.

Regarding waste inventory, the analytes of concern for the HTI Project are antimony, arsenic, barium, cadmium, chromium, lead, silver, nitrate, nitrite, <sup>241</sup>Am, <sup>60</sup>Co, <sup>137</sup>Cs, <sup>239/240</sup>Pu, <sup>79</sup>Se, <sup>90</sup>Sr,

and  $^{99}\text{Tc}$  (Banning 1998). Appendix B provides a detailed description of the analytical results. The HTI DQO does not establish notification limits for the individual analytes.

Data were obtained for all required analytes (Esch 1998); however, the  $^{79}\text{Se}$  data are considered suspect and should be used with caution. During the liquid scintillation analysis, energy was observed in the area where  $^{79}\text{Se}$  would be expected. However, because no actual peak was observed, it is believed that the energy was not from  $^{79}\text{Se}$  but was instead caused by interference from high levels of  $^{137}\text{Cs}$  in the waste. Consequently, a mean for  $^{79}\text{Se}$  was not derived.

Preliminary results from the required leach study are presented in Appendix B. Deviations were required from the original work plan. The leach test was to be performed on a composite of material from auger and light-duty utility arm samples. Because of delays in deploying the light-duty utility arm, the decision was made to proceed with the leach test on a composite of the auger samples only (Schreiber 1998c). A determination was also made to restrict the composite material to only the riser 9G augers because of concerns about the representativeness of the riser 3A auger samples. Results for all required analytes were obtained except  $^{79}\text{Se}$ . As with the analyses on the individual auger samples, no notification limits are established by the HTI DQO.

## 2.2 SAFETY SCREENING

The data needed to screen the waste in tank 241-AX-104 for potential safety problems are documented in *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995). These potential safety problems are exothermic conditions in the waste, flammable gases in the waste and/or tank headspace, and criticality conditions in the waste. Each condition is addressed separately below.

### 2.2.1 Exothermic Conditions (Energetics)

The first requirement outlined in the safety screening DQO (Dukelow et al. 1995) is to ensure there are not sufficient exothermic constituents (organic or ferrocyanide) in tank 241-AX-104 to pose a safety hazard. The safety screening DQO required the waste sample profile be tested for energetics every 24 cm (9.5 in.) to determine whether the energetics exceeded the safety threshold limit. The threshold limit for energetics is 480 J/g on a dry weight basis.

The auger sampling and analysis plan (SAP) (Schreiber 1998a) required a differential scanning calorimetry (DSC) analysis to assess energetics. A DSC analysis was performed on the riser 3A augers. However, a DSC analysis could not be performed on the riser 9G samples because of the high dose rates associated with the auger samples. Consequently, Schreiber (1998b) directed that the DSC analysis be replaced by a total organic carbon (TOC) analysis by furnace oxidation. Because no ferrocyanide is expected in the tank based on the process history, TOC would be the source of any energetics. Therefore, a TOC analysis provides equivalent results to the DSC analysis.

A threshold limit of 45,000  $\mu\text{g C/g}$  (dry weight) has been established for TOC concentration (Adams 1998a). Upper limits (ULs) to 95 percent confidence intervals for the analytical sample means are used for comparison to the threshold. For the riser 9G samples, all TOC results were below detection levels, so no confidence intervals were calculated. After converting the riser 3A

TOC data to a dry-weight basis, the highest mean was 2,930  $\mu\text{g C/g}$ , and the highest 95 percent confidence interval UL was 5,550  $\mu\text{g C/g}$ . Therefore, all results and confidence interval ULs were at least eight times less than the notification limit. No exothermic behavior was observed in the DSC analysis on the riser 3A samples. Based on the DSC and TOC data, energetics is not a concern for tank 241-AX-104.

### 2.2.2 Flammable Gas

Data from two different events are available to evaluate the flammable gas issue for tank 241-AX-104. Headspace measurements using a combustible gas meter were taken from riser 3A before the November 1997 auger sampling. Flammable gas was not detected in the tank headspace (results were 0 percent of the lower explosive limit [LEL]). Combustible gas meter readings were also taken through riser 3A prior to the January 1997 vapor sampling event. No flammable gas was detected as the results were reported as zero percent of the LEL. Both of these measurements are well below the safety screening DQO limit of 25 percent of the LEL. Data for these vapor phase measurements are presented in Section B2.2.

### 2.2.3 Criticality

The safety screening DQO threshold for criticality, based on total alpha activity, is 1 g/L. Because total alpha activity is measured in  $\mu\text{Ci/g}$  instead of g/L, the 1-g/L limit is converted into units of  $\mu\text{Ci/g}$  by assuming that all alpha decay originates from  $^{239}\text{Pu}$ . The safety threshold limit then becomes 1 g  $^{239}\text{Pu}$  per liter of waste. Using the only analytically determined density value available (1.80 g/mL [from the 1977 historical sample]), 1 g/L of  $^{239}\text{Pu}$  is converted to 34.2  $\mu\text{Ci/g}$  of alpha activity.

Total alpha activity was measured for the riser 3A samples. However, before this analysis was performed on the riser 9G augers, revisions to the tank 241-AX-104 analytical requirements were made (Schreiber 1998c). One of the changes made in Schreiber (1998c) was to delete the total alpha activity measurements for the riser 9G samples because a  $^{239/240}\text{Pu}$  analysis was already required.

The highest total alpha activity sample mean from the riser 3A samples was 0.00125  $\mu\text{Ci/g}$ . The highest 95 percent confidence interval UL was 0.00170  $\mu\text{Ci/g}$ , well below the safety screening limit. The highest individual  $^{239/240}\text{Pu}$  result for the riser 9G augers was 8.61  $\mu\text{Ci/g}$  from auger 97-AUG-003. The mean from this sample was 7.6  $\mu\text{Ci/g}$ , which produced a 95 percent confidence interval UL of 14.0  $\mu\text{Ci/g}$ . The overall tank mean was 6.66  $\mu\text{Ci/g}$ , and the UL to the 95 percent confidence interval was 18.6  $\mu\text{Ci/g}$ . Because all of these results were below the 34.2  $\mu\text{Ci/g}$  threshold, criticality is not a concern for this tank. Appendix C contains the method used to calculate confidence limits.

If total alpha activity had been measured on the individual riser 9G auger samples, the results likely would have exceeded the safety screening threshold, as indicated by data from a total alpha activity analysis on a composite of riser 9G material. The composite analysis yielded a result of 41.5  $\mu\text{Ci/g}$  of total alpha activity. This result was not unexpected because of the large

concentrations of  $^{241}\text{Am}$ . However, because the true radionuclide of concern regarding criticality is  $^{239}\text{Pu}$ , and all of the  $^{239/240}\text{Pu}$  results were below safety screening limits, criticality is not a concern.

## 2.3 ORGANIC COMPLEXANTS

The data required to support the organic complexants issue are documented in *Memorandum of Understanding for the Organic Complexant Safety Issue Data Requirements* (Schreiber 1997). Usually, energetics by DSC and sample moisture analyses by thermogravimetry are conducted to address the organic complexants issue. However, because of the high dose rates associated with riser 9G samples, DSC could only be performed on the riser 3A samples. A TOC analysis by furnace oxidation was used in place of DSC for the riser 9G samples. The high dose rates also precluded a thermogravimetric analysis. Instead, gravimetry was used to determine moisture content. The moisture content data are needed only for converting the DSC and TOC results to a dry-weight basis.

As discussed in Section 2.2.1, energetics is not a concern for tank 241-AX-104. No exothermic behavior was observed in the DSC analysis for the riser 3A samples, and the dry-weight TOC results and 95 percent confidence interval ULs from these samples were at least eight times below the 45,000  $\mu\text{g C/g}$  notification limit. All TOC results from the riser 9G samples were below detection limits.

The organic complexants safety issue was closed in December 1998 (Owendoff 1998).

## 2.4 ORGANIC SOLVENTS SAFETY SCREENING

The data required to support the organic solvent screening issue are documented in *Data Quality Objective to Support Resolution of the Organic Solvent Safety Issue* (Meacham et al. 1997). The DQO requires tank headspace samples be analyzed for total nonmethane organic compounds to determine whether the organic extractant pool in the tank is a hazard. The purpose of this assessment is to ensure that an organic solvent pool fire or ignition of organic solvents cannot occur.

The January 1997 vapor samples had a total nonmethane organic hydrocarbon concentration of  $< 0.080 \text{ mg/m}^3$ . This converts to an estimated organic solvent pool size of  $0.03 \text{ m}^2$  (Huckaby and Sklarew 1997), below the limit of  $1 \text{ m}^2$ .

The organic solvents issue is expected to be closed in 1999.

## 2.5 OTHER TECHNICAL ISSUES

### 2.5.1 Hazardous Vapor Screening

Vapor samples were taken to address the requirements of *Data Quality Objectives for Tank Hazardous Vapor Safety Screening* (Osborne and Buckley 1995). However, hazardous vapor

screening is no longer an issue because headspace vapor (sniff) tests are required for the safety screening DQO (Dukelow et al. 1995), and the toxicity issue was closed for all tanks (Hewitt 1996).

## 2.5.2 Tank Waste Heat Load

A factor in assessing tank safety is the heat generation and temperature of the waste. Heat is generated in the tanks from radioactive decay. An estimate of the tank heat load based on the 1997 auger sampling event was derived using the radionuclide data, as shown in Table 2-1. Note that to provide the most conservative estimate, a density of 1.8 g/mL and a volume of 28.4 kL (7.5 kgal) were used when converting concentrations to inventories.

The best-basis inventory radionuclide data yielded a heat load estimate of 18,100 W (61,800 Btu/hr) (note that this value is biased high because the best-basis radionuclide inventories are decay corrected to January 1, 1994). This estimate is above the 11,700 W (40,000 Btu/hr) threshold that separates high- and low-heat-load tanks. Tank 241-AX-104 is not currently considered a high-heat-load tank (Hanlon 1998). Other heat load estimates of 2,960 W (10,100 Btu/hr) (based on process history [Agnew et al. 1997a]) and 4,220 W (14,400 Btu/hr) (based on tank headspace temperatures [Kummerer 1995]) indicate that the tank may not be a high-heat-load tank. Because of these conflicting heat load estimates and the uncertainty surrounding the waste volume, a definitive categorization regarding heat load cannot be made at this time. Additional data that will aid in making the determination are expected to be obtained during light duty utility arm (LDUA) sampling in the first or second quarter of 1999.

Table 2-1. Projected Heat Load.

Analyte	Inventory (Ci) <sup>1</sup>	W/Ci	W
<sup>241</sup> Am	972	0.0328	31.9
<sup>60</sup> Co	334	0.0154	5.14
<sup>137</sup> Cs	63,300	0.00472	299
<sup>154</sup> Eu	1,870	0.00898	16.8
<sup>155</sup> Eu	1,700	7.23E-04	1.23
<sup>239</sup> Pu	286	0.0305	8.72
<sup>240</sup> Pu	54.5	0.0306	1.67
<sup>90</sup> Sr	2.64E+06	0.00670	17,700
Total			18,100

Note:

<sup>1</sup>Best-basis inventory values



## 2.6 SUMMARY

The results of all analyses performed to address potential safety issues showed that primary analytes did not exceed safety decision threshold limits. The heat load categorization remains unresolved. A summary of the technical issues is presented in Table 2-2.

Table 2-2. Summary of Technical Issues.

Issue	Sub-issue	Result
Tank 241-AX-104 waste inventory and leach study	n/a	A waste inventory was derived for all required analytes except $^{79}\text{Se}$ based on the auger analytical results. Results from the leach study were obtained for all required analytes except $^{79}\text{Se}$ .
Safety screening	Energetics	The riser 9G TOC results were below detection limits. No exotherms were observed for the riser 3A samples, and the dry-weight TOC results and 95 percent confidence interval ULs for these samples were at least eight times below the 45,000 $\mu\text{g C/g}$ limit.
	Flammable gas	Results from two separate combustible gas meter readings of the tank headspace were below the 25% LEL threshold (both 0% of the LEL).
	Criticality	All results and 95 percent confidence interval ULs for total alpha (riser 3A data) and $^{239/240}\text{Pu}$ (riser 9G data) were below 34.2 $\mu\text{Ci/g}$ .
Organic complexants <sup>1</sup>	Safety categorization (safe)	No exothermic behavior was observed in the riser 3A samples, and all TOC results and 95 percent confidence interval ULs were below 45,000 $\mu\text{g C/g}$ .
Organic solvents	Solvent pool size	Organic pool size is estimated to be 0.03 $\text{m}^2$ , well below the limit of 1 $\text{m}^2$ .

Note:

<sup>1</sup>The organic complexants safety issue was closed in December 1998 (Owendoff 1998).

### 3.0 BEST-BASIS STANDARD INVENTORY ESTIMATE

Chemical and radionuclide inventory estimates are generally derived from one of three sources of information: 1) sample analysis and sample derived inventory estimates, 2) component inventories predicted by the Hanford defined waste (HDW) model based on process knowledge and historical tank transfer information, or 3) a tank-specific process estimate based on process flowsheets, reactor fuel data, essential materials records, or comparable sludge layers and sample information from other tanks.

An effort is currently underway to provide waste inventory estimates that will serve as the standard characterization data for various waste management activities. As part of this effort, a survey and analysis of various sources of information relating to the chemical and radionuclide component inventories in tank 241-AX-104 was performed that included:

1. Data from auger samples obtained in 1997 (Esch 1998). Only data from the riser 9G samples (97-AUG-003 and 97-AUG-004) were used in the assessment.
2. Data from a solids sample obtained in 1977 after the first sluicing campaign (Starr 1977).
3. Component inventory estimates provided by the HDW model (Agnew et al. 1997a).

Based on this analysis, a best-basis inventory was developed. The 1997 auger samples were used to generate estimates for most of the chemical and radionuclide components in this waste. This waste mostly consists of PUREX high-level waste from Al-clad fuel. The best-basis inventory for tank 241-AX-104, based on a waste volume of 28.4 kL (7.5 kgal), is presented in Tables 3-1 and 3-2. The inventory values reported in Tables 3-1 and 3-2 are subject to change. Refer to the Tank Characterization Database for the most current inventory values.

Once the best-basis inventories were determined, the hydroxide inventory was calculated by performing a charge balance with the valences of other analytes. This charge balance approach is consistent with that used by Agnew et al. (1997a).

Best-basis tank inventory values are derived for 46 key radionuclides (as defined in Section 3.1 of Kupfer et al. [1998]), all decayed to a common report date of January 1, 1994. Often, waste sample analyses have only reported  $^{90}\text{Sr}$ ,  $^{137}\text{Cs}$ ,  $^{239/240}\text{Pu}$ , and total uranium (or total beta and total alpha), while other key radionuclides such as  $^{60}\text{Co}$ ,  $^{99}\text{Tc}$ ,  $^{129}\text{I}$ ,  $^{154}\text{Eu}$ ,  $^{155}\text{Eu}$ , and  $^{241}\text{Am}$  have been infrequently reported. For this reason, it has been necessary to derive most of the 46 key radionuclides by computer models. These models estimate radionuclide activity in batches of reactor fuel, account for the split of radionuclides to various separations plant waste streams, and track their movement with tank waste transactions. These computer models are described in Kupfer et al. (1998), Section 6.1, and in Watrous and Wootan (1997). Model-generated values for radionuclides in any of 177 Hanford Site tanks are reported in the HDW Rev. 4 model results (Agnew et al. 1997a). The best-basis value for any one analyte may be either a model result or a sample- or engineering assessment-based result if available. For a discussion of typical error between model derived-values and sample-derived values, see Kupfer et al. (1998), Section 6.1.10.

Table 3-1. Best-Basis Inventory Estimates for Nonradioactive Components in Tank 241-AX-104 (Effective December 10, 1998).

Analyte	Total Inventory (kg)	Basis (S, M, E or C) <sup>1</sup>	Comment
Al	2,700	S	Starr (1977) = 1,890
Bi	0	E	No process history of Bi
Ca	619	S	Starr (1977) = 729
Cl	16.0	S	
TIC as CO <sub>3</sub>	557	M/E	HDW model concentration value scaled to 28.4 kL (7.5 kgal)
Cr	29.5	S	Starr (1977) = 90.1
F	5.16	S	
Fe	13,900	S	Starr (1977) = 8,420
Hg	0	E	Simpson (1998)
K	88.4	S/E	Upper bounding estimate
La	75.1	S	
Mn	240	S	Starr (1977) = 132
Na	2,200	S	Starr (1977) = 1,950
Ni	433	S	Starr (1977)
NO <sub>2</sub>	115	S	Starr (1977) = 108
NO <sub>3</sub>	2,340	S	Starr (1977) = 158
OH <sub>TOTAL</sub>	19,600	C	Based on charge balance
Pb	474	S	
PO <sub>4</sub>	128	S	Based on ICP. Starr (1977) = 297
Si	44.4	S	QC problems with 1997 value. Starr (1977) = 1,980
SO <sub>4</sub>	242	S	Based on ICP. Starr (1977) = 341
Sr	48.7	S	
TOC	114	S/E	Upper bounding estimate
U <sub>TOTAL</sub>	168	S	Starr (1977) = 0.121
Zr	202	S	

Notes:

ICP = inductively coupled plasma spectroscopy

TIC = total inorganic carbon

<sup>1</sup>S = Sample-based, M = HDW model-based (Agnew et al. 1997a), E = engineering assessment-based, and C = calculated by charge balance; includes oxides as hydroxides, not including CO<sub>3</sub>, NO<sub>2</sub>, NO<sub>3</sub>, PO<sub>4</sub>, SO<sub>4</sub>, and SiO<sub>3</sub>.

Table 3-2. Best-Basis Inventory Estimates for Radioactive Components in Tank 241-AX-104, Decayed to January 1, 1994. (Effective December 10, 1998) (2 sheets)

Analyte	Total Inventory (Ci)	Basis (S, M, or E) <sup>1</sup>	Comment
<sup>3</sup> H	3.38	M	
<sup>14</sup> C	0.63	M	
<sup>59</sup> Ni	3.12	M	
<sup>60</sup> Co	334	S	Sarr (1977)
<sup>63</sup> Ni	313	M	
<sup>79</sup> Se	0.0500	S	<sup>79</sup> Se suffered from quality control failures
<sup>90</sup> Sr	2.64 E+06	S	
<sup>90</sup> Y	2.64 E+06	S	Referenced to <sup>90</sup> Sr
<sup>93m</sup> Nb	10.2	M	
<sup>93</sup> Zr	13.6	M	
<sup>99</sup> Tc	95.3	S	
<sup>106</sup> Ru	0.0101	M	
<sup>113m</sup> Cd	58.2	M	
<sup>125</sup> Sb	3.90	M	
<sup>126</sup> Sn	4.61	M	
<sup>129</sup> I	0.00864	M	
<sup>134</sup> Cs	0.207	M	
<sup>137</sup> Cs	63,300	S	
<sup>137m</sup> Ba	59,900	S	Referenced to <sup>137</sup> Cs
<sup>151</sup> Sm	11,000	M	
<sup>152</sup> Eu	3.36	M	
<sup>154</sup> Eu	1,870	S	Sarr (1977)
<sup>155</sup> Eu	1,700	S	Sarr (1977)
<sup>226</sup> Ra	1.96 E-04	M	
<sup>227</sup> Ac	0.00106	M	
<sup>228</sup> Ra	1.77 E-09	M	
<sup>229</sup> Th	2.77 E-07	M	
<sup>231</sup> Pa	0.00238	M	
<sup>232</sup> Th	1.60 E-10	M	
<sup>232</sup> U	4.67E-06	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>233</sup> U	1.10E-07	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution

Table 3-2. Best-Basis Inventory Estimates for Radioactive Components in Tank 241-AX-104, Decayed to January 1, 1994. (Effective December 10, 1998) (2 sheets)

Analyte	Total Inventory (Ci)	Basis (S, M, or E) <sup>1</sup>	Comment
<sup>234</sup> U	0.0576	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>235</sup> U	0.00240	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>236</sup> U	0.00157	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>237</sup> Np	0.00954	M	
<sup>238</sup> Pu	11.0	S/M	Based on <sup>239</sup> Pu and HDW isotopic distribution
<sup>238</sup> U	0.0561	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>239</sup> Pu	286	S/M	Based on <sup>239/240</sup> Pu and HDW isotopic distribution
<sup>240</sup> Pu	54.5	S/M	Based on <sup>239/240</sup> Pu and HDW isotopic distribution
<sup>241</sup> Am	972	S	
<sup>241</sup> Pu	785	S/M	Based on <sup>239</sup> Pu and HDW isotopic distribution
<sup>242</sup> Cm	0.888	S/M	Based on <sup>241</sup> Am and HDW isotopic distribution
<sup>242</sup> Pu	0.00454	S/M	Based on <sup>239</sup> Pu and HDW isotopic distribution
<sup>243</sup> Am	0.0298	S/M	Based on <sup>241</sup> Am and HDW isotopic distribution
<sup>243</sup> Cm	0.0682	S/M	Based on <sup>241</sup> Am and HDW isotopic distribution
<sup>244</sup> Cm	2.10	S/M	Based on <sup>241</sup> Am and HDW isotopic distribution

Note:

<sup>1</sup>Sample-based, M =HDW model-based (Agnew et al. 1997a), and E = engineering assessment-based.

#### 4.0 RECOMMENDATIONS

The results of all analyses performed to address potential safety issues showed that the TOC concentration, headspace flammable gas concentration, and  $^{239/240}\text{Pu}$  concentration were below their respective safety decision threshold limits. Vapor samples showed the estimated organic pool size was well below the safety limit of  $1 \text{ m}^2$ .

Table 4-1 summarizes the Project Hanford Management Contractor (PHMC) TWRS Program review status and acceptance of the sampling and analysis results reported in this TCR. All issues required to be addressed by sampling and analysis are listed in column 1 of Table 4-1. Column 2 indicates by "yes" or "no" whether issue requirements were met by the sampling and analysis performed. Column 3 indicates concurrence and acceptance by the program in PHMC/TWRS responsible for the applicable issue. A "yes" in column 3 indicates that no additional sampling or analyses are needed. Conversely, a "no" indicates additional sampling or analysis may be needed to satisfy issue requirements.

Sampling and analysis for the tank 241-AX-104 waste characterization DQO (Banning 1998) have been only partially performed; only one tank stratum, the tank floor, has been sampled to date. Waste on the remaining two tank strata (the tank walls/hardware and the tank ceiling) will be sampled in the future using the LDUA.

Results from the 1997 auger samples are considered adequate for assessing the issues of the safety screening DQO. As discussed in Section 2.0, data from the riser 3A auger samples were not included in derivation of tank means in order to provide the most radiologically conservative waste inventory. However, although likely different from a majority of the tank solids, the riser 3A augers do provide a profile of the waste underneath the riser. Consequently, the results were used in combination with the riser 9G data to perform the safety screening assessment. Use of two vertical profiles satisfies the sampling requirement of the safety screening DQO.

Table 4-1. Acceptance of Tank 241-AX-104 Sampling and Analysis.

Issue	Sampling and Analysis Performed	TWRS/PHMC Program Acceptance
Tank 241-AX-104 waste characterization DQO	Partial	No (more sampling required)
Organic complexants memorandum of understanding <sup>1</sup>	Yes	Yes
Organic solvents DQO <sup>2</sup>	Yes	Yes
Safety screening DQO	Yes	Yes

Notes:

<sup>1</sup>The organic complexants safety issue was closed in December 1998.

<sup>2</sup>The organic solvents issue is expected to be closed in 1999.

Table 4-2 summarizes the status of PHMC TWRS Program review and acceptance of the evaluations and other characterization information contained in this report. Column 1 lists the different evaluations performed in this report. Column 2 shows whether issue evaluations have been completed or are in progress. Column 3 indicates concurrence and acceptance with the evaluation by the program in PHMC/TWRS that is responsible for the applicable issue. A "yes" indicates that the evaluation is completed and meets all issue requirements.

The evaluation for the Tank 241-AX-104 Waste Characterization DQO (Banning 1998) can only partially be completed at this time because further sampling is needed. Also, the leach study is still in progress, with only preliminary results published at this time.

Table 4-2. Acceptance of Evaluation of Characterization Data and Information for Tank 241-AX-104.

Issue	Evaluation Performed	TWRS/PHMC Program Acceptance
Tank 241-AX-104 waste characterization DQO	Partial; in progress	n/a
Organic complexants memorandum of understanding <sup>1</sup>	Yes	Yes
Organic solvents DQO <sup>2</sup>	Yes	Yes
Safety screening DQO	Yes	Yes

Notes:

<sup>1</sup>The organic complexants safety issue was closed in December 1998.

<sup>2</sup>The organic solvents issue is expected to be closed in 1999.

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## **APPENDIX A**

### **HISTORICAL TANK INFORMATION**

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## APPENDIX A

### HISTORICAL TANK INFORMATION

Appendix A describes tank 241-AX-104 based on historical information. For this report, historical information includes information about the fill history, waste types, surveillance, or modeling data about the tank. This information is necessary for providing a balanced assessment of sampling and analytical results.

This appendix contains the following information:

- **Section A1.0:** Current tank status, including the current waste levels and the tank stabilization and isolation status
- **Section A2.0:** Information about the tank design
- **Section A3.0:** Process knowledge about the tank, the waste transfer history, and the estimated contents of the tank based on modeling data
- **Section A4.0:** Surveillance data for tank 241-AX-104, including surface-level readings, temperatures, and a description of the waste surface based on photographs
- **Section A5.0:** Appendix A references.

#### A1.0 CURRENT TANK STATUS

According to the Waste Tank Summary Report, tank 241-AX-104 contained an estimated 26.5 kL (7 kgal) of noncomplexed waste as of September 30, 1998 (Hanlon 1998). This waste volume was estimated using a manual tape surface-level gauge and an auto ENRAF<sup>1</sup> (the auto ENRAF™ was installed in October 1996). A separate in-depth volume assessment using in-tank measurements (temperature, gamma dose level, and magnetometer readings) estimated the volume to be between 18.9 kL (5 kgal) and 28.4 kL (7.5 kgal) (Reich 1997). Table A1-1 shows the volumes of the waste phases found in the tank. A volume of 28.4 kL (7.5 kgal) is assumed for this report.

Tank 241-AX-104 was declared an assumed leaker in 1977 and removed from service in 1978. It was interim stabilized in 1981 and intrusion prevention was completed in December 1982. The tank is passively ventilated and is not on the Watch List (Public Law 101-510).

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<sup>1</sup> ENRAF is a trademark of ENRAF Corporation, Houston, Texas.

Table A1-1. Tank Contents Status Summary.

Waste Type	kL (kgal)
Total waste	28.4 (7.5) <sup>1</sup>
Supernatant	0 (0) <sup>2</sup>
Sludge	28.4 (7.5) <sup>1</sup>
Saltcake	0 (0) <sup>2</sup>
Drainable interstitial liquid	0 (0) <sup>2</sup>
Drainable liquid remaining	0 (0) <sup>2</sup>
Pumpable liquid remaining	0 (0) <sup>2</sup>

## Notes:

<sup>1</sup>Reich (1997) estimates between 18.9 kL (5 kgal) and 28.4 kL (7.5 kgal).<sup>2</sup>Hanlon (1998).**A2.0 TANK DESIGN AND BACKGROUND**

The AX Tank Farm was constructed from 1963 to 1964 in the 200 East Area of the Hanford Site. The AX Tank Farm contains four 100 series tanks. These tanks have a capacity of 3,785 kL (1,000 kgal) and a diameter of 22.9 m (75.0 ft). The 241-AX Tank Farm was designed for boiling or self-concentrating waste (for a 5- to 10-year boiling period) with a maximum fluid temperature of 121 °C (250 °F) (Leach and Stahl 1997). Because the tanks are designed specifically for boiling waste, airlift circulators were installed to control waste temperatures.

The single-shell tanks in the 241-AX Tank Farm are constructed of 30-cm (1-ft)- thick reinforced concrete with a 6.4-mm (0.25-in.) mild carbon steel liner on the bottom and sides and a 38-cm (1.25-ft)-thick domed concrete top. They have a flat bottom with a 15-cm (6-in.) radius knuckle and a 9.2-m (30.3-ft) operating depth. A grid of drain slots exits below the tank liner of each tank. There are no cascade overflow lines between the tanks in the 241-AX Tank Farm. The tanks are covered with approximately 2 m (6 ft) of overburden.

Tank 241-AX-104 has 35 risers that penetrate the tank vapor space according to the drawings and engineering change notices. The risers range in diameter from 10 cm (4 in.) to 107 cm (42 in.). Table A2-1 shows numbers, diameters, and descriptions of the risers. A plan view that depicts the riser and nozzle configuration is shown as Figure A2-1. Risers 3A, 7C, 7D, 9E, 9G, 16B, and 16C are tentatively available for sampling (Lipnicki 1997). Riser 3A is 41 cm (16 in.) in diameter, while riser 9G is 15 cm (6 in.) in diameter. A tank cross section showing the approximate waste level along with a schematic of the tank equipment is in Figure A2-2.

Table A2-1. Riser Descriptions for Tank 241-AX-104.<sup>1,2,3,4</sup> (2 sheets)

Number	Diameter		Description and Comments
	cm	in.	
1A	86	34	Sludge sluice, weather covered
1B	86	34	Sludge sluice, weather covered
3A*	41	16	B-222 observation port, bench mark
4	51	20	Vapor outlet, below grade
5A	30	12	Pump mount, weather covered
5B	30	12	Pump mount, weather covered
6*	10	4	Tank pressure, below grade
7A	10	4	Temperature probe
7B	10	4	Temperature probe
7C	10	4	Drain
7D	10	4	Drain
8A	15	6	Dry well
8B	15	6	Dry well
8C	15	6	Dry well
8D	15	6	Dry well
8E	15	6	Dry well
8F	15	6	Dry well
8G	15	6	Dry well
9A*	15	6	Spare port, blind flange (Engineering Change Notice 642930)
9B	15	6	ENRAF <sup>TM</sup> surface-level gauge
9C	15	6	Temperature probe
9D*	15	6	Drain
9E	15	6	Air filter
9F*	15	6	Sludge measurement port
9G	15	6	Spare port, blind flange (Engineering Change Notice 619193)
10	10	4	Drain, weather covered
12	10	4	Leak detection pit drain, below grade
13A	10	4	Temperature probe, weather covered
13B	10	4	Temperature probe, weather covered
13C	10	4	Temperature probe, weather covered
14	107	42	Sludge sluice, weather covered
15	10	4	Condensate addition, below grade
16A	10	4	Flange



Table A2-1. Riser Descriptions for Tank 241-AX-104.<sup>1,2,3,4</sup> (2 sheets)

Number	Diameter		Description and Comments
	cm	in.	
16B	10	4	Flange
16C	10	4	Flange

## Notes:

\*Denotes risers tentatively available for sampling (Lipnicki 1997)

<sup>1</sup>Alstad (1993)

<sup>2</sup>Tran (1993)

<sup>3</sup>Vitro Engineering Corporation (1986)

<sup>4</sup>22 airlift circulators (shown in Figure A2-1 as AC1-22) and 3 structural thermocouples (used to monitor temperatures within the concrete shell of the tank [shown in Figure A2-1 as 11A-C]) also exist.

Figure A2-1. Riser Configuration for Tank 241-AX-104.

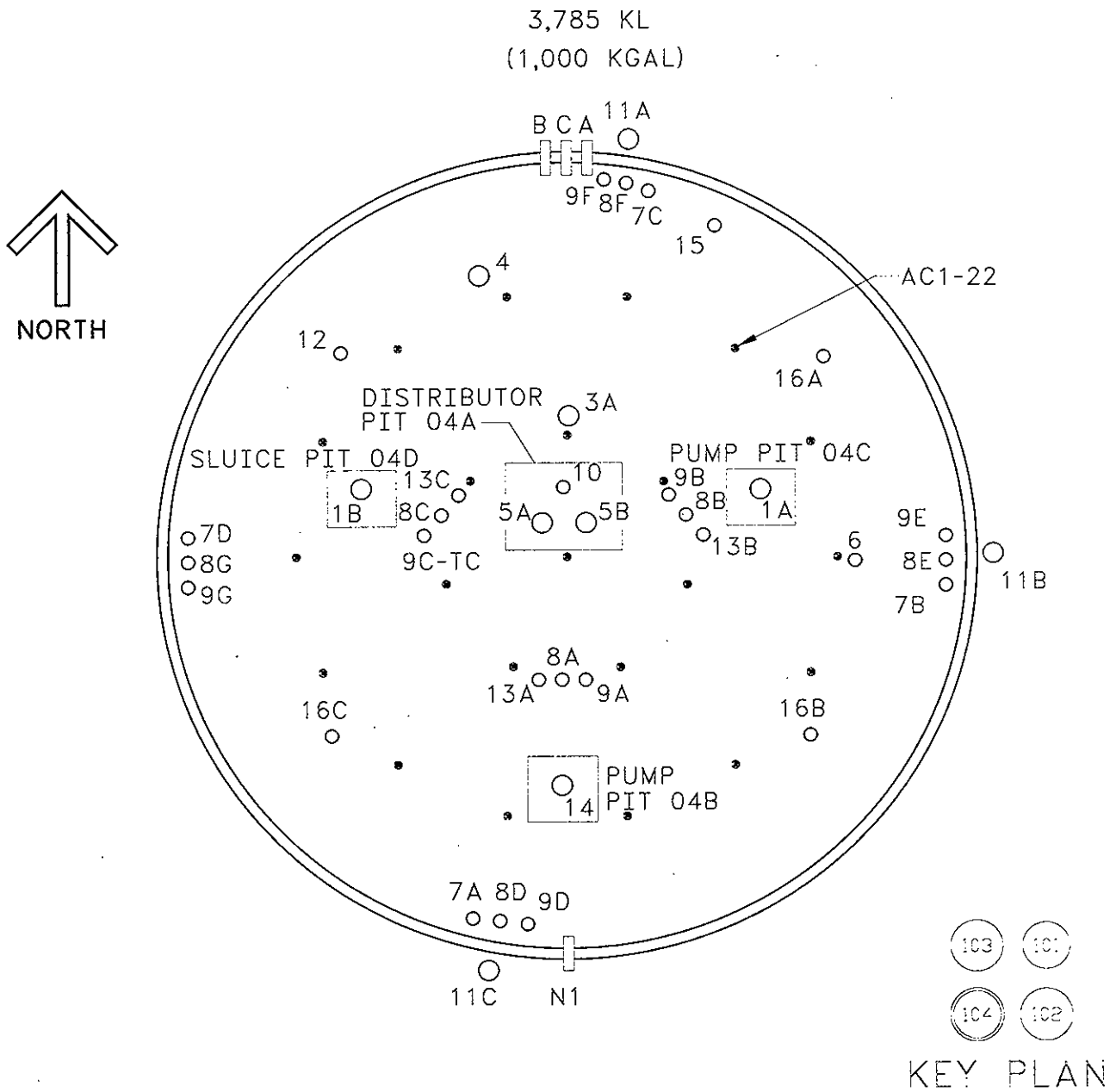
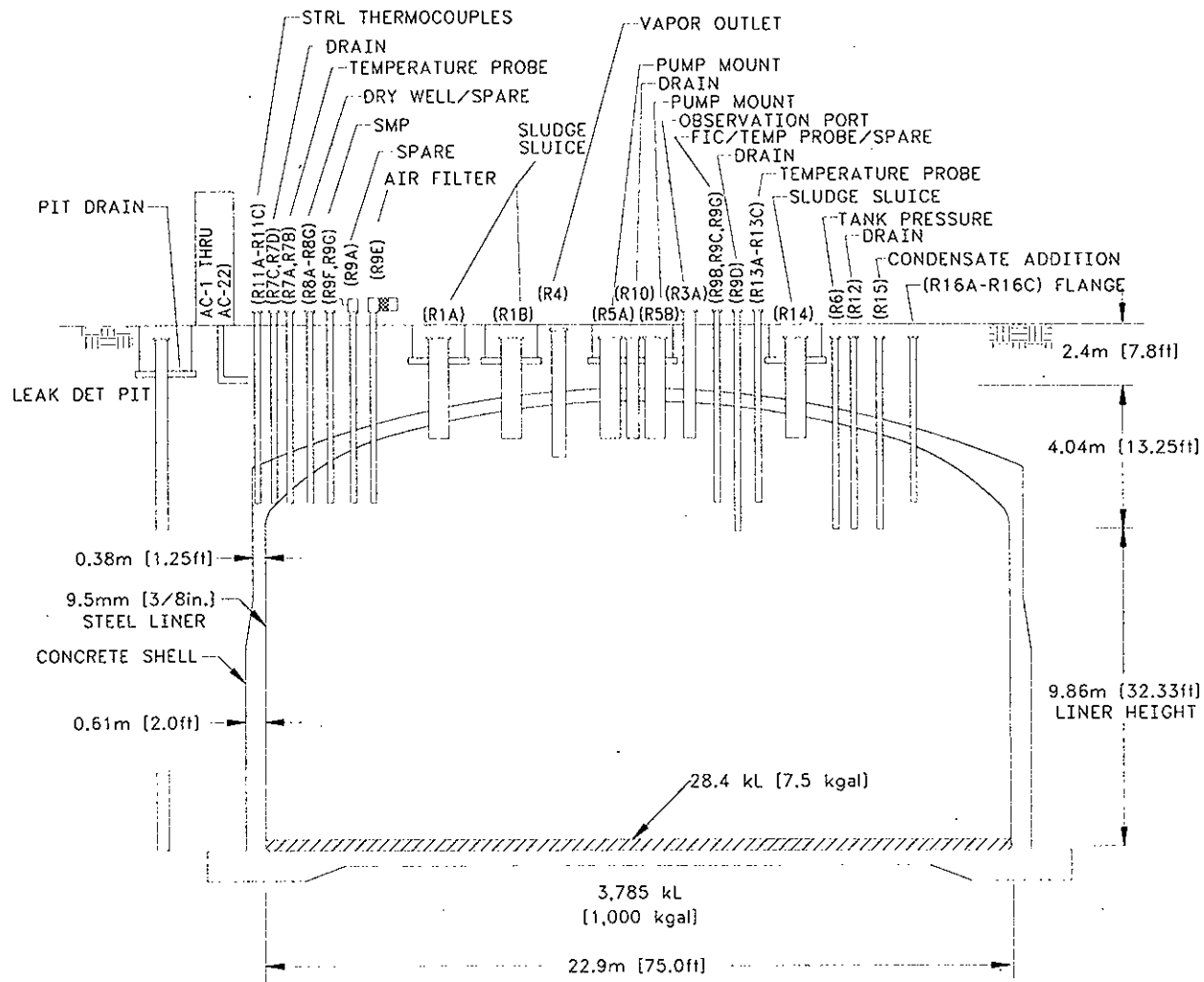


Figure A2-2. Tank 241-AX-104 Cross Section and Schematic.



### A3.0 PROCESS KNOWLEDGE

The sections below 1) provide information about the transfer history of tank 241-AX-104, 2) describe the process wastes that made up the transfers, and 3) estimate the current tank contents based on transfer history.

#### A3.1 WASTE TRANSFER HISTORY

Table A3-1 summarizes the waste transfer history of tank 241-AX-104 (Agnew et al. 1997b). The tank entered service in the third quarter of 1965 when it received water from miscellaneous sources. From the fourth quarter of 1965 through the first quarter of 1969, intermittent transfers were made to and from tank 241-A-102. Beginning in the third quarter of 1966 and continuing through the second quarter of 1969, the tank received PUREX high- and low-level waste. Organic wash waste (containing low solids) from PUREX was received in the first two quarters of 1968. During the second quarter of 1968 and the first quarter of 1969, several small amounts of B Plant waste (from which strontium had been extracted) were transferred into the tank. From 1970 until the end of the tank's active service life, transfers of supernatant waste were made both to and from a variety of other single-shell tanks. According to Agnew et al. (1997b), the last supernatant transfer occurred in the fourth quarter of 1977.

The majority of the tank solids were removed during a sluicing campaign in the second and third quarters of 1977. The intent of the campaign was to remove the high-heat-generating strontium and cesium isotopes. A volume assessment following the sluicing campaign revealed that additional waste removal was needed because the volume and thickness of the waste remaining in the tank could still potentially produce thermal hot spots (Reich 1997). A second sluicing campaign was initiated in the beginning of 1978 and continued until mid-April of that year (Rodenhizer 1987). Upon conclusion of this campaign, no further waste removal was required.

Currently, the waste in tank 241-AX-104 is classified as noncomplexed. The tank was declared an assumed leaker in 1977, with an estimated leakage volume of 30 kL (8 kgal) (see Hanlon [1998] for a description of the method used to derive the leakage volume). The tank was administratively interim stabilized in 1981, and intrusion prevention was completed in 1982.

Table A3-1 presents a summary of the major transfers into and out of tank 241-AX-104.

Table A3-1. Tank 241-AX-104 Major Transfers.<sup>1</sup>

Transfer Source	Transfer Destination	Waste Type	Time Period	Estimated Waste Volume	
				kL	kgal
Miscellaneous	---	Water	1965 - 1974	4,686	1,238
---	241-A-102	Supernatant	1965 - 1969	9,361	2,473
241-A-102	---	Supernatant	1966	1,260	333
PUREX	---	P2; PL1	1966 - 1969	8,006	2,115
PUREX	---	OWW3	1968	897	237
B Plant	---	B	1968 - 1969	185	49
---	241-A-101	Supernatant	1970	1,500	396
---	241-A-106	Supernatant	1972 - 1973	613	162
---	241-AX-103	Supernatant	1972 - 1976	7,052	1,863
241-A-104	---	Supernatant	1972 - 1974	4,879	1,289
241-AX-103	---	Supernatant	1974 - 1976	337	89
002-AR	---	Supernatant	1976	34	9
---	241-C-105	Supernatant	1976	886	234
241-A-102	---	Supernatant	1976 - 1977	2,030	536
---	241-A-102	Supernatant	1977	2,920	772

## Notes:

- B = acid waste from PUREX acidified waste, processed through B Plant for strontium extraction  
 OWW3 = organic wash waste generated from 1968-72  
 P2 = PUREX high-level waste generated from 1963-67  
 PL1 = PUREX low-level waste generated from cell drainage and vessel cleanout

<sup>1</sup>Agnew et al. (1997b)

### A3.2 HISTORICAL ESTIMATION OF TANK CONTENTS

The historical transfer data used for this estimate are from the following sources.

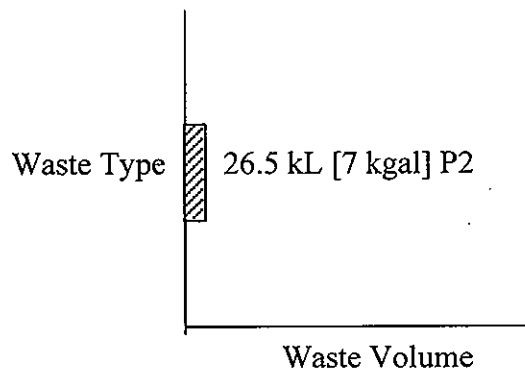
- *Waste Status and Transaction Record Summary: WSTRS, Rev. 4*, (Agnew et al. 1997b) is a tank-by-tank quarterly summary spreadsheet of waste transactions.
- *Hanford Tank Chemical and Radionuclide Inventories: HDW Model Rev. 4* (Agnew et al. 1997a) contains the HDW list and waste type compositions, the supernatant mixing model (SMM), the tank layer model (TLM), and the HDW model tank inventory estimates.
- The HDW list is comprised of approximately 50 waste types defined by concentration for major analytes/compounds for sludge and supernatant layers.
- The TLM defines the solid layers in each tank using waste composition and waste transfer information.

- The SMM is a subroutine within the HDW model that calculates the volume and composition of certain supernatant blends and concentrates.

Using these records, the TLM defines the solid layers in each tank. The SMM uses information from the Waste Status and Transaction Record Summary (WSTRS), the TLM, and the HDW list to describe the supernatants and concentrates in each tank. Together, theWSTRS, TLM, SMM, and HDW list determine the inventory estimate for each tank. These model predictions are considered estimates that require further evaluation using analytical data.

Based on Agnew et al. (1997a), tank 241-AX-104 contains only a layer of 26.5 kL (7 kgal) of PUREX high-level (P2) waste. Figure A3-1 is a graphical representation of the estimated waste type and volume for the tank layer. The HDW model predicts that tank 241-AX-104 contains greater than 1 weight percent (wt%) of iron, hydroxide, sodium, silicon, nitrite, and carbonate, and between 1 and 0.1 wt% of nickel, calcium, sulfate, and ammonia. High levels of  $^{90}\text{Sr}$  and  $^{137}\text{Cs}$  are predicted, and the estimated amounts of  $^{241}\text{Am}$  and  $^{239/240}\text{Pu}$  are at higher concentrations than many of the Hanford Site tanks. Table A3-2 shows the historical estimate of the expected waste constituents and their concentrations.

Figure A3-1. Tank Layer Model.



Note: The current tank volume is assumed to be 28.4 kL (7.5 kgal) for this report.

Table A3-2. Hanford Defined Waste Model Tank Inventory Estimate: Analytes.<sup>1,2,3</sup> (2 sheets)

Total Inventory Estimate							
Physical Properties				-95 CI	-67 CI	+67 CI	+95 CI
Total waste	35,600 kg; 7 kgal						
Heat load	2.96 kW (10,100 Btu/hr)			2.61	2.86	3.03	3.07
Bulk density	1.34 g/mL			1.19	1.29	1.38	1.40
Water wt% <sup>4</sup>	61.5			57.6	59.3	65.0	71.7
TOC wt% C (wet)	0			0	0	0	0
Chemical Constituents	M	ppm	kg	-95 CI (M)	-67 CI (M)	+67 CI (M)	+95 CI (M)
Na <sup>+</sup>	4.03	68,900	2,450	0.428	2.83	4.78	5.33
Al <sup>3+</sup>	0	0	0	0	0	0	0
Fe <sup>3+</sup> (total Fe)	2.96	1.23E+05	4,380	2.86	2.93	2.97	2.98
Cr <sup>3+</sup>	0.00650	252	8.96	0.00246	0.00449	0.00854	0.0105
Bi <sup>3+</sup>	0	0	0	0	0	0	0
La <sup>3+</sup>	0	0	0	0	0	0	0
Hg <sup>2+</sup>	0	0	0	0	0	0	0
Zr (as ZrO(OH) <sub>2</sub> )	0	0	0	0	0	0	0
Pb <sup>2+</sup>	0	0	0	0	0	0	0
Ni <sup>2+</sup>	0.0581	2,540	90.4	0.00123	0.0345	0.0699	0.0766
Sr <sup>2+</sup>	0	0	0	0	0	0	0
Mn <sup>4+</sup>	0	0	0	0	0	0	0
Ca <sup>2+</sup>	0.243	7,250	258	0.00558	0.125	0.302	0.335
K <sup>+</sup>	0.00458	133	4.75	0.00174	0.00317	0.00602	0.00741
OH <sup>-</sup>	9.14	1.16E+05	4,120	8.64	8.97	9.26	9.34
NO <sub>3</sub> <sup>-</sup>	6.17E-17	2.85E-12	1.01E-13	7.32E-18	1.96E-17	2.81E-16	5.59E-15
NO <sub>2</sub> <sup>-</sup>	0.587	20,100	715	0.222	0.405	0.771	0.948
CO <sub>3</sub> <sup>2-</sup>	0.243	10,900	386	0.00558	0.125	0.302	0.335
PO <sub>4</sub> <sup>3-</sup>	0	0	0	0	0	0	0
SO <sub>4</sub> <sup>2-</sup>	0.129	9,230	328	0.0489	0.0891	0.170	0.209
Si (as SiO <sub>3</sub> <sup>2-</sup> )	1.52	31,800	1,130	0.0283	1.08	1.75	1.94
F <sup>-</sup>	0	0	0	0	0	0	0
Cl <sup>-</sup>	0.0211	556	19.8	0.00798	0.0146	0.0277	0.0341
C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> <sup>3-</sup>	0	0	0	0	0	0	0
EDTA <sup>4-</sup>	0	0	0	0	0	0	0
HEDTA <sup>3-</sup>	0	0	0	0	0	0	0
Glycolate <sup>-</sup>	0	0	0	0	0	0	0
Acetate <sup>-</sup>	0	0	0	0	0	0	0

Table A3-2. Hanford Defined Waste Model Tank Inventory Estimate: Analytes.<sup>1,2,3</sup> (2 sheets)

Total Inventory Estimate							
Chemical Constituents	M	ppm	Kg	-95 CI (M)	-67 CI (M)	+67 CI (M)	+95 CI (M)
Oxalate <sup>2-</sup>	0	0	0	0	0	0	0
DBP	0	0	0	0	0	0	0
Butanol	0	0	0	0	0	0	0
NH <sub>3</sub>	0.270	3,410	122	0.106	0.190	0.352	0.431
Fe(CN) <sub>6</sub> <sup>4-</sup>	0	0	0	0	0	0	0

## Notes:

CI = confidence interval

DBP = dibutyl phosphate

<sup>1</sup>Agnew et al. (1997a)<sup>2</sup>Unknowns in tank solids inventory are assigned by TLM.<sup>3</sup>HDW model inventory predictions have not been validated and should be used with caution.<sup>4</sup>Water weight percent is derived from the difference of density and total dissolved species.Table A3-3. Hanford Defined Waste Model Tank Inventory Estimate: Radionuclides.<sup>1,2,3</sup> (3 sheets)

Total Inventory Estimate							
Radiological Constituents	Ci/L	μCi/g	Ci	-95 CI (Ci/L)	-67 CI (Ci/L)	+67 CI (Ci/L)	+95 CI (Ci/L)
<sup>3</sup> H	1.28E-04	0.0950	3.38	1.76E-05	5.96E-05	2.22E-04	3.38E-04
<sup>14</sup> C	2.38E-05	0.0177	0.630	9.00E-06	1.64E-05	3.12E-05	3.84E-05
<sup>59</sup> Ni	1.18E-04	0.0877	3.12	2.49E-06	7.00E-05	1.42E-04	1.55E-04
<sup>63</sup> Ni	0.0118	8.81	313	2.50E-04	7.03E-03	0.0142	0.0156
<sup>60</sup> Co	3.28E-05	0.0244	0.868	1.24E-05	2.26E-05	4.30E-05	3.17E-04
<sup>79</sup> Se	1.11E-04	0.0830	2.95	1.02E-05	8.53E-05	1.29E-04	1.47E-04
<sup>90</sup> Sr	16.2	12,000	4.28E+05	14.5	15.7	16.4	16.5
<sup>90</sup> Y	16.2	12,000	4.29E+05	14.5	15.7	16.4	16.5
<sup>93</sup> Zr	5.12E-04	0.381	13.6	9.11E-06	3.81E-04	6.02E-04	6.87E-04
<sup>93m</sup> Nb	3.84E-04	0.286	10.2	3.00E-05	2.93E-04	4.47E-04	5.07E-04
<sup>99</sup> Tc	1.69E-04	0.125	4.47	6.38E-05	1.16E-04	2.21E-04	2.72E-04
<sup>106</sup> Ru	3.82E-07	2.84E-04	0.0101	1.29E-07	3.16E-07	4.27E-07	4.70E-07
<sup>113m</sup> Cd	0.00220	1.63	58.2	4.41E-05	0.00141	0.00273	0.00325
<sup>125</sup> Sb	1.47E-04	0.110	3.90	5.57E-05	1.02E-04	1.93E-04	2.38E-04
<sup>126</sup> Sn	1.74E-04	0.129	4.61	2.20E-05	1.35E-04	2.01E-04	2.27E-04



Table A3-3. Hanford Defined Waste Model Tank Inventory Estimate: Radionuclides.<sup>1,2,3</sup>  
(3 sheets)

Total Inventory Estimate							
Radiological Constituents (Cont'd)	Ci/L	μCi/g	Ci	-95 CI (Ci/L)	-67 CI (Ci/L)	+67 CI (Ci/L)	+95 CI (Ci/L)
<sup>129</sup> I	3.26E-07	2.43E-04	0.00864	1.23E-07	2.25E-07	4.28E-07	5.27E-07
<sup>134</sup> Cs	7.81E-06	0.00581	0.207	2.96E-06	5.39E-06	1.03E-05	1.26E-05
<sup>137</sup> Cs	0.621	463	16,500	0.235	0.429	0.816	1.00
<sup>137m</sup> Ba	0.588	438	15,600	0.223	0.406	0.772	0.950
<sup>151</sup> Sm	0.416	309	11,000	0.0612	0.324	0.478	0.538
<sup>152</sup> Eu	1.27E-04	0.0944	3.36	1.25E-04	1.26E-04	1.28E-04	1.29E-04
<sup>154</sup> Eu	0.00731	5.44	194	2.36E-04	7.46E-04	0.0118	0.0161
<sup>155</sup> Eu	0.00945	7.03	250	0.00928	0.00937	0.00955	0.00964
<sup>226</sup> Ra	7.39E-09	5.51E-06	1.96E-04	4.26E-09	6.59E-09	7.96E-09	8.49E-09
<sup>228</sup> Ra	6.69E-14	4.98E-11	1.77E-09	6.57E-14	6.63E-14	6.76E-14	6.82E-14
<sup>227</sup> Ac	3.99E-08	2.97E-05	0.00106	2.17E-08	3.52E-08	4.31E-08	4.62E-08
<sup>231</sup> Pa	8.98E-08	6.68E-05	0.00238	1.67E-09	6.36E-08	1.08E-07	1.25E-07
<sup>229</sup> Th	1.04E-11	7.77E-09	2.77E-07	1.03E-11	1.04E-11	1.06E-11	1.06E-11
<sup>232</sup> Th	6.04E-15	4.49E-12	1.60E-10	2.28E-15	4.17E-15	7.93E-15	9.75E-15
<sup>232</sup> U	8.39E-12	6.24E-09	2.22E-07	3.18E-12	5.79E-12	1.10E-11	1.36E-11
<sup>233</sup> U	1.98E-13	1.47E-10	5.24E-09	7.49E-14	1.37E-13	2.60E-13	3.20E-13
<sup>234</sup> U	1.03E-07	7.70E-05	0.00274	3.91E-08	7.14E-08	1.36E-07	1.67E-07
<sup>235</sup> U	4.31E-09	3.21E-06	1.14E-04	1.63E-09	2.98E-09	5.67E-09	6.97E-09
<sup>236</sup> U	2.82E-09	2.10E-06	7.47E-05	1.07E-09	1.95E-09	3.71E-09	4.56E-09
<sup>238</sup> U	1.01E-07	7.49E-05	0.00267	3.81E-08	6.95E-08	1.32E-07	1.63E-07
<sup>237</sup> Np	3.60E-07	2.68E-04	0.00954	1.36E-07	2.49E-07	4.73E-07	5.82E-07
<sup>238</sup> Pu	9.42E-05	0.0701	2.50	7.72E-05	8.99E-05	9.73E-05	1.00E-04
<sup>239</sup> Pu	0.00244	1.82	64.7	0.00200	0.00233	0.00252	0.00260
<sup>240</sup> Pu	4.66E-04	0.347	12.3	3.82E-04	4.44E-04	4.81E-04	4.95E-04
<sup>241</sup> Pu	0.00671	4.99	178	0.00550	0.00640	0.00693	0.00713
<sup>242</sup> Pu	3.88E-08	2.89E-05	0.00103	3.18E-08	3.70E-08	4.01E-08	4.12E-08
<sup>241</sup> Am	0.00384	2.86	102	0.00232	0.00345	0.00411	0.00437
<sup>243</sup> Am	1.18E-07	8.77E-05	0.00312	7.12E-08	1.06E-07	1.26E-07	1.34E-07
<sup>242</sup> Cm	3.51E-06	0.00261	0.0929	3.44E-06	3.48E-06	3.54E-06	3.58E-06
<sup>243</sup> Cm	2.69E-07	2.01E-04	0.00714	2.64E-07	2.67E-07	2.72E-07	2.75E-07
<sup>244</sup> Cm	8.28E-06	0.00617	0.219	3.22E-06	6.98E-06	9.18E-06	1.00E-05

Table A3-3. Historical Tank Inventory Estimate: Radionuclides.<sup>1,2,3</sup> (3 sheets)

Total Inventory Estimate							
Totals	<i>M</i>	µg/g	kg	-95 CI ( <i>M</i> or g/L)	-67 CI ( <i>M</i> or g/L)	+67 CI ( <i>M</i> or g/L)	+95 CI ( <i>M</i> or g/L)
Pu	0.0414 (g/L)	----	1.10	0.0339	0.0395	0.0428	0.0440
U	0.00127	224	7.99	4.80E-04	8.75E-04	0.00166	0.00205

## Notes:

<sup>1</sup>Agnew et al. (1997a)<sup>2</sup>Unknowns in tank solids inventory are assigned by TLM.<sup>3</sup>HDW model inventory predictions have not been validated and should be used with caution.

## A4.0 SURVEILLANCE DATA

Tank 241-AX-104 surveillance consists of surface-level measurements, temperature monitoring inside the tank (waste and headspace), and leak detection well (dry well) monitoring for radioactivity outside the tank. Surveillance data provide the basis for determining tank integrity. Solid surface-level measurements indicate physical changes in and consistencies of the solid layers of a tank. Dry wells located around the tank perimeter may show increased radioactivity resulting from leaks.

## A4.1 SURFACE-LEVEL READINGS

Daily surface-level readings are currently taken using an ENRAF<sup>TM</sup> gauge. This gauge was installed through riser 9B in October 1996 and readings were initially taken manually. Daily readings using the automatic mode began in September 1997. The ENRAF<sup>TM</sup> measurements have been stable, ranging from 3.53 cm (1.39 in.) to 4.47 cm (1.76 in.). On November 11, 1998, the surface-level was 3.63 cm (1.43 in.) as measured by the ENRAF<sup>TM</sup> gauge. This measurement equates to a waste depth of 14.9 kL (3.93 kgal). However, this estimate is based on the assumption that the waste surface is level, and tank photographs and other in-tank measurements have demonstrated that the surface level can vary up to five inches (Reich 1997). Before installation of the ENRAF<sup>TM</sup> gauge, a manual tape was used to monitor the surface level. Readings from the manual tape, located in riser 9A, are available from January 1981 to October 1996. Figure A4-1 is a depiction of the level history through 1995 (Brevick et al. 1997). A graph of the surface level measurements since January 1996 taken from the Surveillance Analysis Computer System is presented in Figure A4-2. The apparent changes shown in Figure A4-2 in the waste level are most likely caused by changes in the level measurement instrumentation and recalibration of the instrumentation, and not by actual changes in the waste level.

Tank 241-AX-104 is categorized as an assumed leaker. Because of the lack of liquid in the tank, no leak detection criterion exists for a decrease in surface level. The surface level increase criterion is 5.1 cm (2.0 in.). The tank does not have a liquid observation well for obtaining information about the quantity of interstitial liquid. However, based on waste surface photographs, in-tank videos, and especially observations of the auger sample material during extrusion and sample handling, no interstitial liquid is anticipated.

Seven dry wells were installed around tank 241-AX-104 in January and February 1975. Three of the dry wells had readings greater than the 50 counts per second background level before 1983. In 1975, increasing radiation was observed in dry well 11-04-11 at a depth of 12 m (40 ft), prompting issuance of a preliminary occurrence report on April 9 of that year. Through subsequent investigation, the source of the contamination was determined to be the tank's 20-in. vapor line at points both above the tank and at the line tied into the 24-in. vessel vent header (Welty 1988). A second occurrence report was issued on January 20, 1976, because of increasing activity in dry well 11-04-08. The occurrence report attributed the activity to further migration of the vapor header leak, although Welty (1988) disputed this conclusion.

Tank 241-AX-104 has a leak detection pit, which was one of the first such pits used at the Hanford Site. As can be seen in Welty (1988), data from the leak detection pit are erratic. No conclusions regarding tank integrity were possible from the leak detection pit data.

#### **A4.2 INTERNAL TANK TEMPERATURES**

Tank 241-AX-104 has only one operable thermocouple tree, located in riser 9C. Other risers previously used for monitoring the waste temperature were 7A, 7B, 13A, 13B, and 13C. Thermocouples in risers 11A, 11B, and 11C were used in the past to measure the temperature of the tank's concrete shell; readings are no longer taken from these thermocouples. Since the first sluicing campaign in the second and third quarters of July 1977, none of the 18 thermocouples on the riser 9C tree have been located in the waste. The closest is positioned 1.07 feet from the tank bottom. Thermocouples 2 through 16 are spaced at 61-cm (2-ft) intervals above thermocouple 1. Thermocouples 17 and 18 are at 122-cm (4-ft) intervals (Tran 1993).

Temperature data are available from the Surveillance Analysis Computer System from October 1976 to July 1, 1998 (the last date measured). The average tank temperature over this period is 36 °C (96 °F), with a high of 117 °C (242 °F) and a low of 20 °C (68 °F). Note that the five highest readings (242, 239, 231, 216, and 216 °F) are from thermocouple 1 when it was still in the waste; the next highest reading is 164 °F. Temperatures are currently taken semi-annually. From July 1, 1997, to July 1, 1998 (the last date measured), temperatures have ranged between 29.2 °C (84.5 °F) and 33.1 °C (91.6 °F), with an average of 31.1 °C (88.0 °F). For plots of the thermocouple readings, refer to the supporting document for the historical tank content estimate (Brevick et al. 1997). Figure A4-3 is a graph of the weekly high temperature.

#### **A4.3 TANK 241-AX-104 PHOTOGRAPHS**

The waste in tank 241-AX-104 has been extensively photographed and videotaped. Photographs were obtained in 1977, 1978, 1981, 1983, 1987, and 1996, and videotaping was done in 1996 and 1997. A complete collage of the 1987 waste surface photos is available in Brevick et al. (1997).

Select video frames from the 1997 videotaping event and historical photographs are available from Reich (1997). The surface color varies from white to reddish-brown. No liquid is currently visible. Dark circles are present under the airlift circulators and other tank equipment. Video data have shown that these circles are actually rings of waste that have fallen off the equipment surfaces. Over 90 percent of the tank knuckle is visible in the videos. In some places, the bare floor adjacent to the knuckle is visible, as well as the weld interfaces between the floor and the knuckle (Reich 1997).

Comparing photographs over the years has yielded discernible changes in the waste surface appearance. The area under riser 3A contained decreasing amounts of liquid in the 1978, 1981, and 1983 photos. In the 1987 photo, the liquid appears to have evaporated and left a red, rust-colored area (Reich 1997).

Figure A4-1. Tank 241-AX-104 Level History Through 1995.

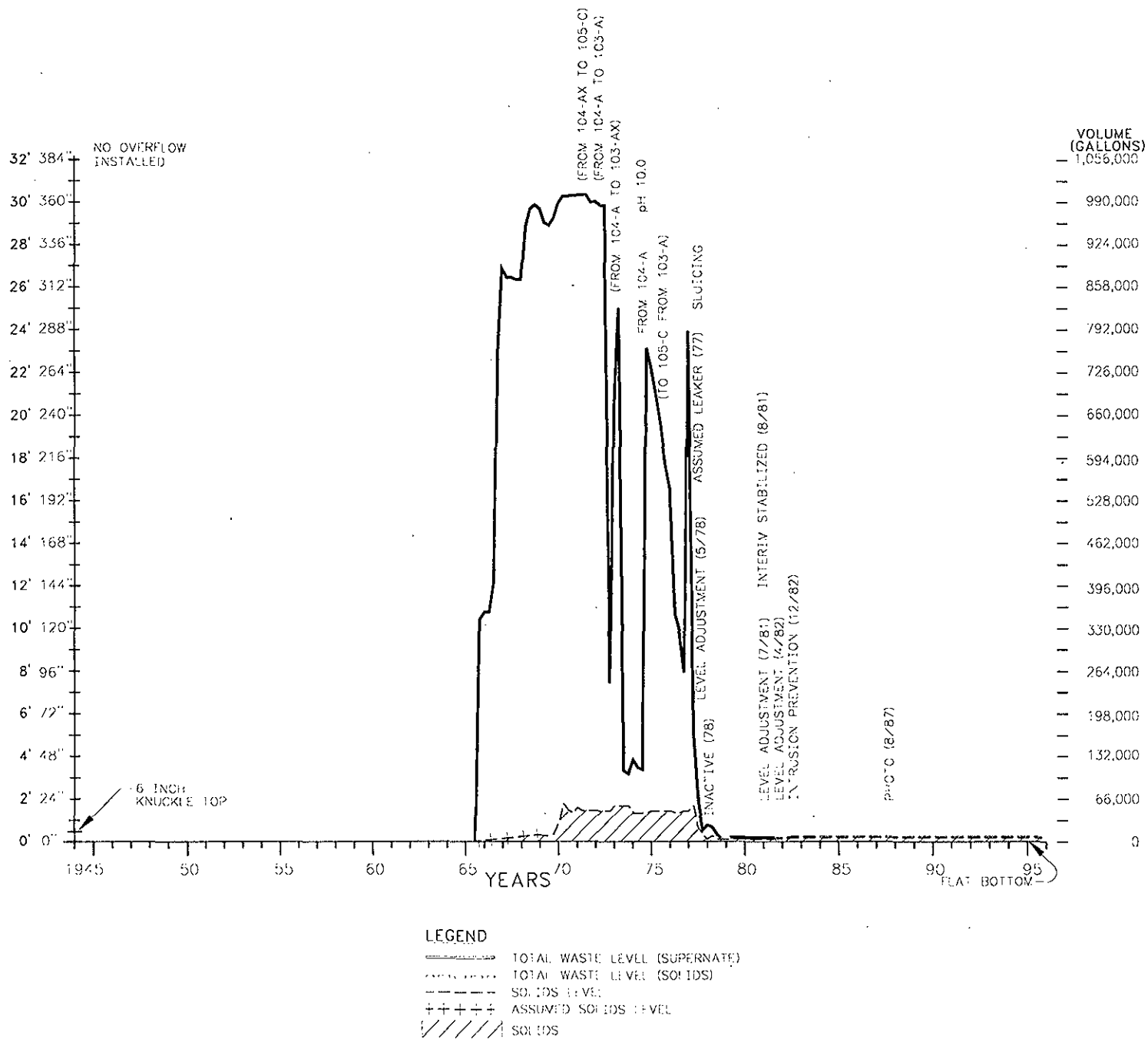


Figure A4-2. Tank 241-AX-104 Level History Since January 1996.

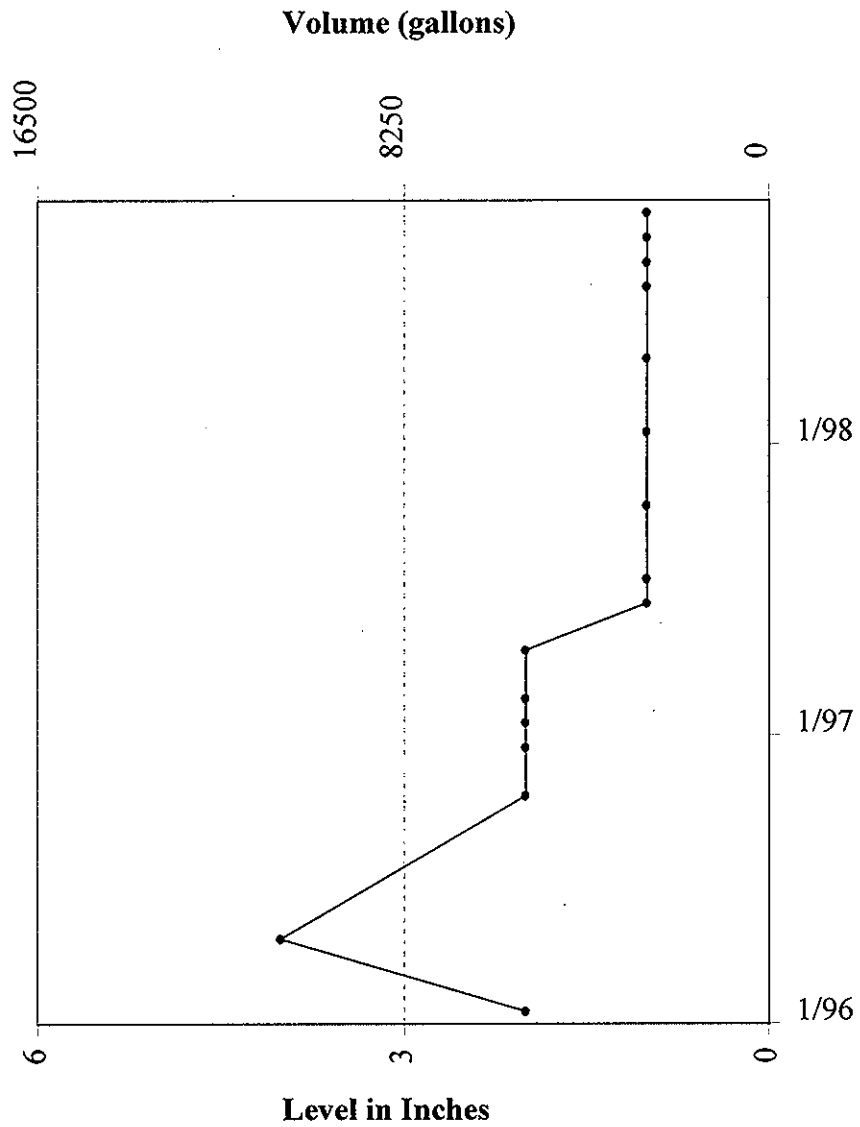
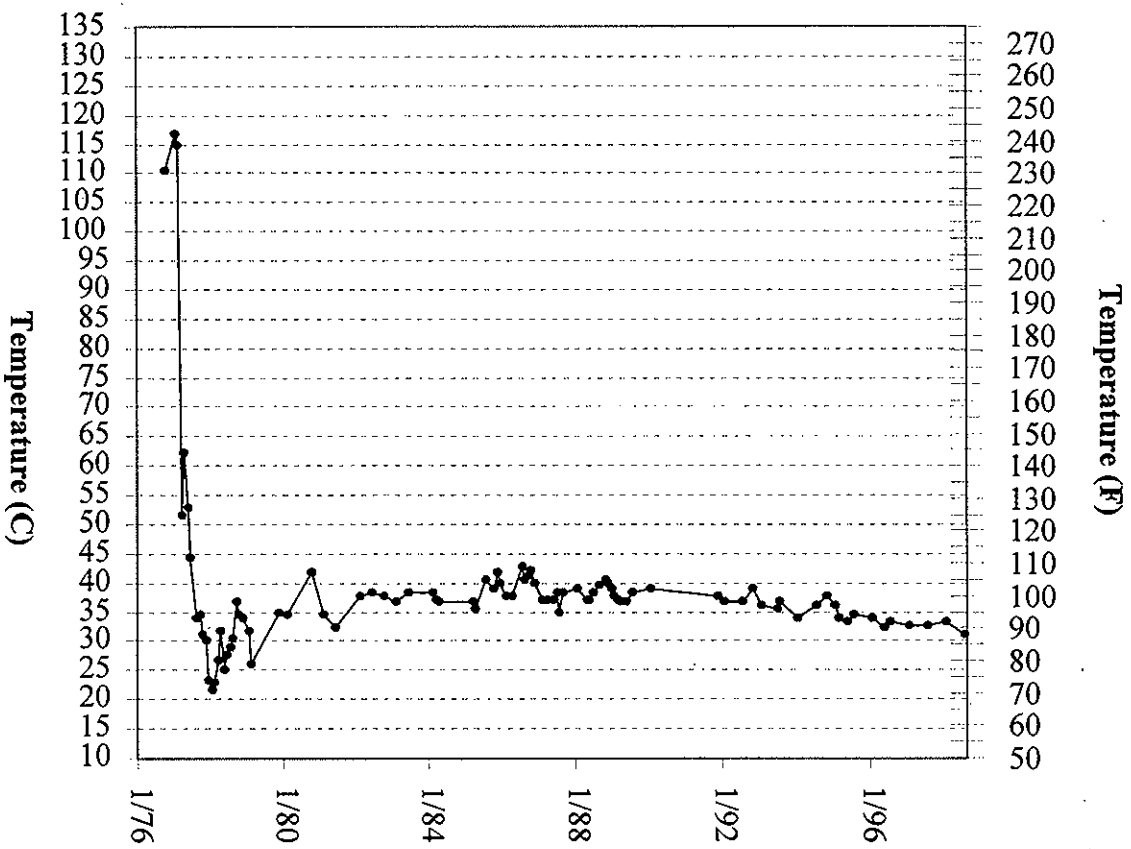


Figure A4-3. Tank 241-AX-104 Temperature Profile.



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**APPENDIX B**

**SAMPLING OF TANK 241-AX-104**

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## APPENDIX B

### SAMPLING OF TANK 241-AX-104

Appendix B provides sampling and analysis information for each known sampling event for tank 241-AX-104 and assesses the 1997 auger sample results. It includes the following:

- **Section B1.0:** Tank Sampling Overview
- **Section B2.0:** Sampling Events
- **Section B3.0:** Assessment of Characterization Results
- **Section B4.0:** Appendix B References.

#### B1.0 TANK SAMPLING OVERVIEW

This appendix describes the sampling and analysis events for tank 241-AX-104. Auger samples were taken in November 1997 to satisfy the requirements of *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995), *Memorandum of Understanding for the Organic Complexant Issue Data Requirements* (Schreiber 1997), and *Tank 241-AX-104 Waste Characterization Data Quality Objective* (Banning 1998) (referred to as the Hanford Tanks Initiative [HTI] DQO). The auger sampling was performed in accordance with *Tank 241-AX-104 Auger Sampling and Analysis Plan* (Schreiber 1998a). Analysis of the auger samples was performed as directed in Schreiber (1998a) and *Tank 241-AX-104 Light Duty Utility Arm Sampling and Analysis Plan* (Schreiber 1998b). Results are discussed in Section B2.1. Vapor sampling was performed in January 1997 to support the Vapor Issue Resolution Program and to satisfy the requirements of *Data Quality Objective for Tank Hazardous Vapor Safety Screening* (Osborne and Buckley 1995). Sampling was performed in accordance with *Vapor Sampling and Analysis Plan* (Buckley 1997) using SUMMA<sup>1</sup> canisters, triple sorbent traps, and sorbent tube trains. Vapor results are presented in Section B2.3. Results from two historical sampling events are reported in this TCR: a core sample obtained in September 1977, and a data set (sampling date unknown) from June 1978. These results are discussed in Section B2.4.

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<sup>1</sup> SUMMA is a trademark of Moleetrics, Inc., Cleveland, Ohio.

**B2.0 SAMPLING EVENTS**

This section describes sampling events and presents analytical results for tank 241-AX-104. The analytical results used to characterize current tank contents were from the 1997 auger samples. Section B2-1 discusses sampling, handling, and analysis of the auger samples. Section B2-2 presents tank vapor headspace measurements. A 1997 vapor sample is discussed in Section B2-3. Historical sample results are presented in Section B2-4. Table B2-1 summarizes the sampling and analytical requirements from the applicable DQOs for the 1997 auger samples and vapor samples.

Table B2-1. Integrated Data Quality Objective Requirements for Tank 241-AX-104. (2 sheets)

<b>Sampling Event</b>	<b>Applicable DQOs</b>	<b>Sampling Requirements</b>	<b>Analytical Requirements</b>
Auger sampling <sup>1</sup>	Safety screening - Energetics - Moisture content - Total alpha - Flammable gas Dukelow et al. (1995)	Samples from a minimum of two risers separated radially to the maximum extent possible.  Combustible gas measurement.	Flammability, energetics, moisture, total alpha activity, density, TOC <sup>2</sup>
	Organic complexant MOU Schreiber (1997)	Two full vertical profiles of the tank waste.	Energetics, moisture, TOC <sup>2</sup>
	HTI Banning (1998)	Obtain samples of each tank stratum (floor, walls/hardware, and dome); auger sampling only addresses the floor stratum.	Anions, cations, radionuclides, fission product screening; leach tests on solids composite <sup>3</sup>

Table B2-1. Integrated Data Quality Objective Requirements for Tank 241-AX-104. (2 sheets)

Sampling Event	Applicable DQOs	Sampling Requirements	Analytical Requirements
Vapor sampling <sup>4</sup>	Hazardous vapor Osborne and Buckley (1995)  Organic solvents Meacham et al. (1997) <sup>5</sup>	SUMMA™ canisters, triple sorbent traps, sorbent tube trains	Flammable gas, organic vapors, permanent gases, total nonmethane hydrocarbons

## Notes:

<sup>1</sup>Schreiber (1998a)<sup>2</sup>TOC is a secondary analyte for both the safety screening and organic complexant DQOs.<sup>3</sup>The leach tests are performed in accordance with Schreiber (1998b).<sup>4</sup>Buckley (1997)<sup>5</sup>The vapor sampling occurred before the release of the organic solvents DQO. The requirements of this DQO have retroactively been applied to the January 1997 data.**B2.1 DESCRIPTION OF 1997 AUGER SAMPLING EVENT**

The intent of the 1997 auger sampling was to obtain two vertical profiles of the tank waste. Vertical profiles are needed to satisfy the safety screening DQO (Dukelow et al. 1995) and the organic complexant memorandum of understanding (Schreiber 1997). Safety screening analyses include: total alpha activity to determine criticality, DSC to ascertain the fuel energy value, and thermogravimetric analysis (TGA) to obtain the total moisture content. In addition, combustible gas meter readings in the tank headspace are performed to measure tank headspace flammability. The safety screening DQO also requires bulk density measurements for use in calculations. The organic complexant MOU requires DSC and TGA. Both documents list TOC (by persulfate oxidation) as a secondary analyte.

The 1997 auger sampling only partially satisfied the requirements of the HTI DQO (Banning 1998). This DQO strives to evaluate the spatial variance in analyte concentration within the tank waste. To meet this objective, the interior of the tank has been divided into three strata for sample collection. The three strata are the floor, walls/hardware, and tank dome. Only the floor stratum can be sampled using the auger sampling method. Sampling of the remaining two strata will be performed in the future using the light duty utility arm (LDUA). The analyses required by the HTI DQO include ICP, ion chromatography (IC), liquid scintillation for <sup>99</sup>Tc and <sup>79</sup>Se,

gamma energy analysis (GEA) for  $^{137}\text{Cs}$ ,  $^{60}\text{Co}$ , and  $^{241}\text{Am}$ , alpha counting for  $^{239/240}\text{Pu}$  and  $^{241}\text{Am}$ , beta counting for  $^{90}\text{Sr}$ , an ICP/mass spectrometry (MS) screen for major fission products, and leach tests. The leach tests were to be performed on a composite of the floor stratum material from the auger and LDUA samples. The leach tests include ICP for seven metals, IC for nitrate and nitrite,  $^{99}\text{Tc}$  by ICP/MS and liquid scintillation,  $^{79}\text{Se}$  by liquid scintillation,  $^{137}\text{Cs}$  and  $^{60}\text{Co}$  by GEA, total alpha and total beta counting, and pH. To provide a baseline for the analyte concentrations, all of the same analyses except pH were to be performed directly on the composite.

Four auger samples were removed from tank 241-AX-104 in November 1997, two each from risers 3A and 9G. The auger samples obtained through riser 3A were taken on November 13, while those removed from riser 9G were taken on November 14 and 21. Sampling was performed in accordance with the auger sampling and analysis plan (SAP) (Schreiber 1998a). Sampling was performed using ten-inch auger samplers. Lithium bromide solution was not added to the drill string during sampling. The chain-of-custody forms for the riser 9G auger samples noted that high levels of contamination were detected under the lids of the shipping casks. A combustible gas meter reading was taken in the tank headspace before sampling. Further discussion of this measurement is provided in Section B2.2.

### B2.1.1 Sample Handling

The four auger samples were shipped to the 222-S Laboratory, where they were extruded and photographed. Table B2-2 presents the extrusion information and sample descriptions. No drainable liquid was collected from any of the segments.

Table B2-2. Extrusion Information and Sample Descriptions. (2 sheets)

Riser	Auger Sample	Weight (g)	Auger Number	Sample Description
3A	97-AUG-001	96.8	Whole	Solids were collected from flutes 1 through 12 as well as the auger liner. Sample appeared as a mixture of fine, light brown powder and darker, coarser material. There were two small clear pieces of plastic or glass that were not collected with the sample.
	97-AUG-002	39.5	Whole	Solids were collected from flutes 1 through 11 as well as the auger liner. Sample appeared as a mixture of fine, light brown powder and darker, coarser material.

Table B2-2. Extrusion Information and Sample Descriptions. (2 sheets)

Riser	Auger Sample	Weight (g)	Auger Number	Sample Description
9G	97-AUG-003	80.8	Whole	Solids were collected from flutes 1 through 18 as well as the auger liner. Solids were dark brown in color and varied from a fine powder to clumps of dried sludge and flakes of crystalline material. Some slightly moist solids adhered to flutes 12 and 13, and were combined with the other material.
	97-AUG-004	36.9	Whole	Material appeared to be on all of the flute edges, but most solids were collected from flutes 1 through 6, as well as the auger liner. Sample varied in appearance from a fine powder to clumps of dried sludge and flakes of crystalline material. There were some small plastic-like pieces that were added to the sample.

**B2.1.1.1 Homogenization.** As described in the HTI DQO (Banning 1998), thorough homogenization of the samples is required to ensure that an analyte of concern is not occluded in matrix mineralogy. The 222-S Laboratory homogenized the samples according to Procedure LO-161-106 (Schreiber 1998a). The work was performed in a hot cell because the volume of sample prepared exceeded 100 g. The samples were homogenized for two minutes, stirred with a spatula for one minute, and then rehomogenized for an additional two minutes.

As required by the HTI DQO (Banning 1998) and directed in the auger SAP (Schreiber 1998a), verification of complete homogenization was performed using a laser ablation/mass spectrometer (LA/MS) instrument. This instrument ablates and analyzes small fractions of solid sample without dissolution. Results from replicate aliquots of the homogenized material must show a relative percent difference (RPD) of 20 percent or less before the sample is considered homogeneous. Because of time constraints, the LA/MS instrument had only demonstrated the ability to achieve a 20 percent relative percent difference (RPD) down to a concentration of 100 ppm. Therefore, the determination of homogeneity was restricted to those analytes with concentrations greater than 100 ppm (Esch 1998).

Sample 97-AUG-003 was the only one that passed the homogeneity test following the first homogenization. The other three samples had failures for a couple of analytes and were homogenized a second time. Unfortunately, quality LA/MS data could not be collected on the



rehomogenized samples because of instrument and data acquisition problems. The homogenization data can be viewed in Attachment 2 of Esch (1998). Further analysis of sample homogeneity by comparing the LA/MS results with the ICP data is also provided in Esch (1998).

A final observation was noted after homogenization. The radioactive content of the riser 9G samples turned the clear glass storage containers black within three days.

**B2.1.1.2 Formation of Composite.** The LDUA SAP instructed the 222-S Laboratory to prepare a composite of waste from the floor stratum using material from both the auger samples and the LDUA samples. Because of delays in deploying the LDUA, Schreiber (1998d) instructed the lab to create the composite for the leach test using material from the auger samples only. Schreiber (1998d) also instructed the lab to use only waste from the riser 9G auger samples in the composite, because the waste in the riser 3A auger samples was deemed unrepresentative of the tank waste (see Section B3.1 for more information on this issue).

Because of insufficient sample material from riser 9G, a composite of 250 g could not be created. Accordingly, Schreiber (1998d) instructed the 222-S Laboratory to generate a composite using as much material as possible. Only enough composite sample material to perform duplicate analyses for  $^{79}\text{Se}$  and  $^{99}\text{Tc}$  was archived.

When forming the composite, material from the riser 9G samples was added in two additions. On the first addition, 35.39 g of 97-AUG-003 was added to 5.77 g of 97-AUG-004. This material was blended, followed by the addition of 36.15 g of 97-AUG-003 and 22.89 g of 97-AUG-004. This brought the final amount of material in the composite from each auger to 71.54 g for 97-AUG-003 and 28.66 g for 97-AUG-004. The weight distribution in the composite is 71.4 weight percent 97-AUG-003 and 28.6 weight percent 97-AUG-004 (Crawford 1998).

The composite material was powdery and dark brown in color. The largest particles were approximately 1.6 mm in diameter and made up less than one-tenth the total volume of the solids. The remaining solid particles were very fine (Crawford 1998). The existence of the larger particles provided further proof that complete homogenization of the individual auger samples had not been achieved following extrusion.

Before dividing into subsamples (an archive sample, an LA/MS homogeneity test sample, and fractions for acid, fusion, and water digestion), the composite material was homogenized (Crawford 1998). A homogeneity check using LA/MS was not performed, however, because of instrument failure.

**B2.1.1.3 Construction of Leach Test.** The leach test was initiated on June 1, 1998, when 87.82 g of solids were added to the leach container, followed by 183.26 g of water. This mixture yielded a 1:2.09 solids to liquids mixture by weight. Stirring began on June 3, 1998, at a rate of 45 rpm using a magnetic stir bar. After 24 hours, the stirring was stopped and observations were

recorded. Within one hour after stirring had ceased, the mixture of solids and liquids had separated into three fractions. The sample appeared to have heavier sludge in the bottom, clear liquid on top of the sludge, and a less dense layer of solids floating on the liquid (Crawford 1998).

Because of problems with unreliable stirring, the magnetic stir bar was replaced with a paddle stirrer that was mounted on the leach container lid. During conversion to the paddle stirrer, 36.03 g of solids and liquids were lost. To determine the amount of each phase that was lost, the residual wet solids remaining after the leach test were dried. The amount of water measured in the wet solids was 61.8 weight percent. Therefore, of the 36.03 g lost, 13.76 g were solids and 22.27 g were water. The resulting water to solids mixture based on this loss was 160.99 g water to 74.06 g solids (a 1:2.2 ratio of solids to liquids) (Crawford 1998).

A sample was removed for analysis (this sample is the 24-hour or 1-day sample). Samples were also removed for analysis after 7 days, 30 days, and 90 days. The temperature and pH were measured at the time of each sampling. Additional temperature measurements were made each week.

### **B2.1.2 Sample Analysis**

The 1997 auger samples were analyzed based on safety screening, organic complexant, and HTI issues. *Tank 241-AX-104 Auger Sampling and Analysis Plan* (Schreiber 1998a) and *Tank 241-AX-104 Light Duty Utility Arm Sampling and Analysis Plan* (Schreiber 1998b) directed the analysis.

**B2.1.2.1 Auger Sampling and Analysis Plan.** The suite of analyses specified in the auger SAP (Schreiber 1998a) included alpha counting for total alpha activity,  $^{239/240}\text{Pu}$ , and  $^{241}\text{Am}$ , DSC for energetics, TGA for water content, gravimetry for bulk density, IC for selected anions (bromide, nitrate, and nitrite), ICP/atomic emission spectroscopy (AES) for selected metals (aluminum, antimony, arsenic, barium, cadmium, chromium, iron, lead, lithium, manganese, nickel, silicon, silver, sodium, and uranium), GEA for  $^{137}\text{Cs}$  and  $^{60}\text{Co}$ , liquid scintillation for  $^{99}\text{Tc}$  and  $^{79}\text{Se}$ , and beta counting for  $^{90}\text{Sr}$ . A screen for major fission products using ICP/MS was also requested.

Several deviations to the auger SAP (Schreiber 1998a) were necessary because of the dry, powdery nature of the samples and the high concentrations of  $^{90}\text{Sr}$  in the two augers from riser 9G (97-AUG-003 and 97-AUG-004). Homogenizing the dry samples generated a fine powder that easily became airborne, increasing the risk for contamination spread. As a result, the samples were not handled outside the hot cell in their dry state. The acid and water digestions were started in the hot cell, and the fusion digestion was performed entirely in the hot cell.

Problems were encountered during completion of the acid digestion outside the hot cell. After receiving the acid-diluted sample from the hot cell, the technician attempted to transfer the entire sample to digestion beakers. Despite repeated rinses, a complete transfer could not be accomplished because the samples appeared to have “clumped” and adhered to the bottom of the sample vials. Because of the high dose rate of the samples, no exceptional efforts were made to recover the remaining material. Because of the difficulties in handling these samples, and concern over radiation exposure for the individual performing the digestion, no redigestion was requested (Esch 1998).

Any direct analyses that could be performed in the hot cell were done so (Schreiber 1998c). The TGA requirement was replaced by gravimetry because a gravimetric analysis can be performed in the hot cell. Differential scanning calorimetry is a direct method and cannot be done in a hot cell. Because of as low as reasonably achievable (ALARA) concerns caused by the substantial amount of radioactivity in the two auger samples from riser 9G, the DSC analysis was deleted from the analytical suite for these two samples. Instead, TOC analysis by furnace oxidation was performed. This method provides energetics data reasonably equivalent to that obtained by DSC, and reduces the risk to laboratory staff by using water digested samples rather than direct samples (Schreiber 1998c).

Schreiber (1998c) also directed that the analysis for total alpha activity be removed from the suite of analyses for the auger samples from riser 9G. Total alpha activity is used as a screening tool for criticality concerns. For this determination, it is assumed that all alpha activity originates from  $^{239}\text{Pu}$ . Because the auger samples were already being analyzed for  $^{239}\text{Pu}$  as required by the auger SAP (Schreiber 1998a), a total alpha analysis was unnecessary. Note that total alpha activity data is available for auger samples 97-AUG-001 and 97-AUG-002 because these samples had already been analyzed by the time the change was made.

Another deviation from the SAP concerned the density measurements. In an effort to conserve sample material, bulk density was not determined on any of the samples.

Although only specific metals and anions were requested during the respective ICP and IC analyses, results for many other metals and anions were obtained. These results are reported on an “opportunistic” basis, and are not subject to quality control (QC) requirements.

**B2.1.2.2 Light-Duty Utility Arm Sampling and Analysis Plan.** The LDUA SAP (Schreiber 1998b) directed analyses on three sample types: the whole LDUA sample; a composite of the auger and LDUA samples; and a leach test sample. However, as of May 1998, sampling using the LDUA system had not yet occurred. Because the composite and leach test analytical data were needed to support other HTI project work, *Revision to Tank 241-AX-104 Leach Test Requirements* (Schreiber 1998d) directed the 222-S Laboratory to perform the analyses specified in the LDUA SAP on the auger samples.

The analytical suites for the composite and leach test samples were nearly identical. Each required alpha counting for total alpha activity, IC for selected anions (bromide, nitrate, and nitrite), ICP/AES for selected metals (antimony, arsenic, barium, cadmium, chromium, lead, lithium, and silver), GEA for  $^{137}\text{Cs}$  and  $^{60}\text{Co}$ , liquid scintillation for  $^{99}\text{Tc}$  and  $^{79}\text{Se}$ , and beta counting for total beta activity. In addition, the LDUA SAP specified a TGA analysis on the composite sample and a pH determination on the leach test sample. As discussed previously, a TGA analysis was not possible because of ALARA concerns. Data for "opportunistic" analytes were obtained during the ICP and IC analyses.

Higher than expected nitrate concentrations were reported for the sample and duplicate from the composited solids. The nitrate concentration was approximately 10 times higher than the estimated values based on the auger results. Other anions were also reported at higher than expected concentrations. To verify these results, the water digestion fraction is currently being reanalyzed (Crawford 1998).

After obtaining results from the composite for  $^{79}\text{Se}$ , the requirements of the LDUA SAP were modified to remove this analysis on the leach test sample. Further discussion on the logic behind this decision is provided in Section B2.1.3.10.

Analyses required by both the auger and LDUA SAPs were either performed directly on the solids or after digestion using water, acid, or fusion. Note that the fusion digestion for  $^{90}\text{Sr}$  was repeated on the individual auger samples because a high concentration of the analyte was detected in the preparation blank on the first preparation. The leach test analyses were performed directly on the liquid samples. All reported analyses were performed following the approved laboratory procedures given in Table B2-3. Tables B2-4 and B2-5 summarize the auger numbers, sample numbers, and analyses performed on each sample.

Table B2-3. Analytical Procedures. (2 sheets)

Analysis	Method	Procedure Number
Energetics	DSC	LA-514-114
Percent water	TGA	LA-564-101
Total alpha activity	Alpha counting	LA-508-101
Flammable gas	Combustible gas analysis	WHC-IP-0030 IH 1.4 and IH-2.1 <sup>2</sup>
TOC	Furnace oxidation	LA-344-105
Metals	ICP/AES	LA-505-161
Anions	IC	LA-533-105
$^{137}\text{Cs}$ , $^{60}\text{Co}$	GEA	LA-548-121
$^{239/240}\text{Pu}$ , $^{241}\text{Am}$	AEA	LA-953-104

Table B2-3. Analytical Procedures.<sup>1</sup> (2 sheets)

Analysis	Method	Procedure Number
<sup>90</sup> Sr	Beta counting	LA-220-101
<sup>79</sup> Se	Liquid scintillation	LA-365-132
<sup>99</sup> Tc	Liquid scintillation	LA-438-101
Screen for major fission products	ICP/MS	LA-506-101
Total beta activity	Beta counting	LA-508-101
PH	pH meter	LA-212-106
Homogenization verification	LA/MS	LT-506-102

Notes:

<sup>1</sup>Schreiber (1998a and 1998b)<sup>2</sup>WHC (1992)

Table B2-4. Sample Analysis Summary for Whole Samples. (2 sheets)

Riser	Auger Number	Sample Number	Preparation Method	Analyses
3A	97-AUG-001	S97T002280	Direct	Percent water (gravimetry)
		S97T002284	Direct	DSC
		S97T002288	Fusion	<sup>90</sup> Sr, AEA
		S97T002301	Acid	<sup>99</sup> Tc and <sup>79</sup> Se, <sup>90</sup> Sr, AEA, ICP, GEA, total alpha, fission product screening
		S97T002305	Water	IC, TOC
		S98T001174	Fusion	<sup>90</sup> Sr
	97-AUG-002	S97T002281	Direct	Percent water (gravimetry)
		S97T002285	Direct	DSC
		S97T002289	Fusion	<sup>90</sup> Sr, AEA
		S97T002302	Acid	<sup>99</sup> Tc and <sup>79</sup> Se, <sup>90</sup> Sr, AEA, ICP, GEA, total alpha, fission product screening
		S97T002306	Water	IC, TOC
		S98T001175	Fusion	<sup>90</sup> Sr

Table B2-4. Sample Analysis Summary for Whole Samples. (2 sheets)

Riser	Auger Number	Sample Number	Preparation Method	Analyses
9G	97-AUG-003	S97T002282	Direct	Percent water (gravimetry)
		S97T002290	Fusion	<sup>90</sup> Sr, AEA
		S97T002303	Acid	<sup>99</sup> Tc and <sup>79</sup> Se, ICP, GEA, fission product screening
		S97T002307	Water	IC, TOC
		S98T001176	Fusion	<sup>90</sup> Sr
	97-AUG-004	S97T002283	Direct	Percent water (gravimetry)
		S97T002291	Fusion	<sup>90</sup> Sr, AEA
		S97T002304	Acid	<sup>99</sup> Tc and <sup>79</sup> Se, ICP, GEA, fission product screening
		S97T002308	Water	IC, TOC
		S98T001177	Fusion	<sup>90</sup> Sr

Table B2-5. Sample Analysis Summary for Composite and Leach Test Samples. (2 sheets)

Sample Type	Sample Number	Preparation Method	Analyses
Composite	S98T001671	Direct	LA/MS (homogeneity test)
	S98T001675	Acid	<sup>79</sup> Se, ICP, ICP/MS
	S98T001677	Fusion	<sup>99</sup> Tc (ICP/MS and liquid scintillation), GEA, total alpha, total beta
	S98T001679	Water	IC
Leach test (liquid)	S98T001768	Direct	ICP, IC, ICP/MS
	S98T001769	Direct	<sup>99</sup> Tc (liquid scintillation), GEA, total alpha, total beta
	S98T001771	Direct	ICP, IC, ICP/MS
	S98T001772	Direct	<sup>99</sup> Tc (liquid scintillation), GEA, total alpha, total beta
	S98T002051	Direct	<sup>99</sup> Tc (ICP/MS), ICP, IC
	S98T002052	Direct	<sup>99</sup> Tc (liquid scintillation), GEA, total alpha, total beta

Table B2-5. Sample Analysis Summary for Composite and Leach Test Samples. (2 sheets)

Sample Type	Sample Number	Preparation Method	Analyses
Leach test (liquid)	S98T002681	Direct	$^{99}\text{Tc}$ (ICP/MS), ICP, IC
Cont'd)	S98T002682	Direct	$^{99}\text{Tc}$ (liquid scintillation), GEA, total alpha, total beta

### B2.1.3 Analytical Results

This section summarizes the sampling and analytical results associated with the November 1997 sampling and analysis of tank 241-AX-104. Table B2-6 shows the location of analytical results within this report. These results are documented in Esch (1998) and Crawford (1998). Note that the composite and leach test data sets are presented separately from the individual auger results.

Substantial differences were observed in analytical results between risers 3A and 9G. One of the most notable differences was the amount of radioactivity in the riser 9G samples, and the relative lack thereof in the riser 3A samples. The process history for tank 241-AX-104 indicates that the tank contents should be highly radioactive. Significant differences were also observed in the ICP and IC data. Because of these differences, uncertainties exist regarding the representativeness of the riser 3A samples to the majority of the tank solids. Consequently, data from the riser 3A auger samples were not used in the calculation of analytical means or inventories. Further discussion regarding this issue is provided in Section B3.1. The riser 3A data were retained in the data tables in Section B2.5 for comparison purposes.

Table B2-6. Analytical Tables. (2 sheets)

Analysis	Table Number
Results of ICP/MS screen	B2-7
Composite and leach test data	B2-8
ICP/AES	B2-14 through B2-50
IC	B2-51 through B2-58
$^{241}\text{Am}$ by alpha energy analysis	B2-59
GEA	B2-60 and B2-61
$^{239/240}\text{Pu}$ by alpha energy analysis	B2-62
$^{79}\text{Se}$ by liquid scintillation	B2-63
$^{89/90}\text{Sr}$ by beta counting	B2-64

Table B2-6. Analytical Tables. (2 sheets)

Analysis	Table Number
<sup>99</sup> Tc by liquid scintillation	B2-65
Total alpha	B2-66
Percent water by TGA	B2-67
TOC by furnace oxidation	B2-68

The QC parameters assessed in conjunction with tank 241-AX-104 samples were standard recoveries, spike recoveries, duplicate analyses (measured by the relative percent difference [RPD] between primary and duplicate subsamples), and blanks. The QC criteria are specified in the auger and LDUA SAPs (Schreiber 1998a and 1998b). Note that QC criteria are not applicable to "opportunistic" analytes. Sample and duplicate pairs, in which any QC parameter was outside these limits, are footnoted in the sample mean column of the Section B2.5 data summary tables with an a, b, c, d, e, or f as follows.

- "a" indicates the standard recovery was below the QC limit.
- "b" indicates the standard recovery was above the QC limit.
- "c" indicates the spike recovery was below the QC limit.
- "d" indicates the spike recovery was above the QC limit.
- "e" indicates the RPD was above the QC limit.
- "f" indicates blank contamination.

In the analytical tables in this section, the "mean" is the average of the result and duplicate value. All values, including those below the detection level (denoted by "<") were averaged. If both sample and duplicate values were nondetected or if one value was detected while the other was not, the mean is expressed as a nondetected value. If both values were detected, the mean is expressed as a detected value.

**B2.1.3.1 Total Alpha Activity.** Analyses for total alpha activity were performed on the individual auger samples, the auger composite, and the leach test sample. As discussed in Section B2.1.2.1, total alpha activity analyses were not performed on the individual riser 9G auger samples. The riser 3A auger samples were prepared by acid digestion, the composite sample was prepared by fusion digestion, and the leach test sample was analyzed directly. The analyses were performed in duplicate. Although a few QC problems were detected, no reruns were requested. For the riser 9G composite sample, the mean alpha activity was 41.5  $\mu\text{Ci/g}$ . By comparison, the results from the riser 3A samples ranged from 8.59E-04 to 0.00125  $\mu\text{Ci/g}$ . Leach test results are discussed in Section B2.1.3.13.



**B2.1.3.2 Gravimetry.** Gravimetric analyses were performed on the individual auger samples in place of TGA. No duplicate result was reported by the 222-S Laboratory for 97-AUG-003 because the result was unacceptable; the gross weight was less than the residual dry weight. No rerun was requested because of the need to conserve sample material for the leach test. Individual sample results ranged from 0.770 to 12.74 percent water. No water content analyses were performed on the composite as required in Schreiber (1998b) because of the high dose rate.

**B2.1.3.3 Differential Scanning Calorimetry.** As described in Section B2.1.2.1, the DSC analysis for the riser 9G auger samples was replaced by a TOC analysis (by furnace oxidation). A discussion of the TOC results is provided in the following subsection. A DSC analysis was performed on the riser 3A auger samples. No exothermic energy was observed in the samples.

**B2.1.3.4 Total Organic Carbon.** Although Schreiber (1998c) requested a TOC analysis for only the riser 9G auger samples, the analysis was performed on all four augers. The TOC subsamples were water digested and measured in duplicate. None of the results exceeded the TOC notification limit of 45,000  $\mu\text{g C/g}$ ; the highest dry weight sample mean was 5,550  $\mu\text{g C/g}$  from auger 97-AUG-002. None of the TOC results in the riser 9G auger samples were above detection limits.

**B2.1.3.5 Inductively Coupled Plasma/Atomic Emission Spectrometry.** The ICP/AES analysis on the individual auger samples was performed in duplicate on acid-digested subsamples. The following metals constituted the requested analytes: aluminum, antimony, arsenic, barium, cadmium, chromium, iron, lead, lithium, manganese, nickel, silicon, silver, sodium, and uranium. All other metals are considered opportunistic. Several QC problems were noted for the ICP subsamples (see Section B3.2). However, no redigestions or reanalyses were requested. The reported detection limits were three to five times higher than those requested in Schreiber (1998a). Lower detection limits could not be attained because of the dilutions required for the high concentrations of iron and sodium in the samples.

Large differences were observed in the ICP results between the auger samples from the two risers. The primary required ICP analytes detected in the riser 9G auger samples were iron, aluminum, sodium, and nickel. Results for these analytes were all greater than 10,000  $\mu\text{g/g}$ . In addition, the riser 9G lead results were close to 10,000  $\mu\text{g/g}$ . For the riser 3A samples, only one analyte, iron, exceeded 10,000  $\mu\text{g/g}$ . All of the aluminum results were below detection limits, half of the sodium results were below detection limits, the nickel results were just over 100  $\mu\text{g/g}$ , and the lead results were approximately 700  $\mu\text{g/g}$ .

Most of the lithium values were below detection levels. The largest detected result was 32.4  $\mu\text{g/g}$ . The lithium results were expected to be low because lithium bromide solution was not used during sampling.

An ICP/AES analysis was also performed on the composite and leach test samples. The composite sample was digested with acid, while the liquid leach test samples were analyzed directly. The required analytes for the ICP analyses were antimony, arsenic, barium, cadmium, chromium, lead, lithium, and silver. Results for the other ICP analytes were reported on an opportunistic basis. The detected ICP results in the composite for the required analytes ranged from 366 µg/g (for silver) to 7,310 µg/g (for lead). Arsenic and antimony concentrations were below detection levels. A discussion of the ICP results from the leaching study is provided in Section B2.1.3.13.

**B2.1.3.6 Ion Chromatography.** Ion chromatography analyses were performed on the individual auger samples, the composite, and the leach test samples. Samples were prepared by water digestion (except the leach test samples, which did not require digestion) and analyzed in duplicate. For all three sample types, the only required analytes were bromide, nitrate, and nitrite. Data for the remaining anions were obtained on an opportunistic basis. Nitrate was the IC analyte present in the largest quantities, ranging from 104,000 to 114,000 µg/g for the riser 3A samples and 26,500 to 65,500 µg/g for the riser 9G samples. For sample 97-AUG-001, the requested detection limit for nitrite could not be met because of the dilution required for the high nitrate concentration. Nitrite was below detection levels for the riser 3A samples but in concentrations between 2,060 and 2,380 µg/g for the riser 9G samples. All bromide results were below detection levels. A discussion of the IC results as related to the leaching study is provided in Section B2.1.3.13.

Higher concentrations of nitrate and nitrite were present in the composite than in the individual auger samples. While the nitrate results for the riser 9G samples ranged between 26,500 and 65,500 µg/g, the composite concentration was 509,000 µg/g. The composite value should be considered conservatively high because of possible matrix effects. High spike values were observed for nitrate, which may have resulted from concentration effects from using smaller spike concentrations in the presence of large concentrations of analyte in the sample. The blank also showed some nitrate present at very low levels (Crawford 1998). For nitrite, the composite mean was 15,700 µg/g, compared with a concentration range of 2,060 to 2,870 µg/g for the riser 9G samples.

**B2.1.3.7 Beta Counting for Strontium-90 and Total Beta Activity.** Because the nitric/hydrochloric acid digestion was not expected to completely digest <sup>90</sup>Sr, only the two auger samples from riser 3A were subjected to this preparation method. All four auger samples were subjected to a fusion digestion and analyzed. Comparing the acid and fusion data for the riser 3A samples demonstrated that the acid digestion did provide incomplete digestion, as the fusion results were slightly higher than the acid results (Crawford 1998). Comparing the <sup>90</sup>Sr data between the riser 3A and riser 9G samples reveals significant concentration differences. The riser 9G samples ranged from 26,300 to 55,300 µCi/g, while those from riser 3A varied between 1.6 and 151 µCi/g.

Substantial blank contamination was found for  $^{90}\text{Sr}$  in the first fusion preparation. Upon repeating the fusion preparation, blank contamination was still present, although at a lower level than previously detected for three of the four auger samples. Section B3.2 provides further discussion regarding this issue. The approximate analysis date was March 1998.

For the composite and leach test samples, values for  $^{90}\text{Sr}$  were derived from the total beta results obtained by beta counting. The composite was analyzed in June 1998 and had a result of 46,600  $\mu\text{Ci/g}$ . Results for the leach test sample are provided in Section B2.1.3.13.

**B2.1.3.8 Gamma Energy Analysis.** For the individual auger samples, a GEA analysis for  $^{137}\text{Cs}$  and  $^{60}\text{Co}$  was performed after an acid digestion. The approximate analysis date was March 1998. Dilutions were required because of the relatively high concentration of  $^{137}\text{Cs}$  in the samples, causing high detection limits for  $^{60}\text{Co}$ . Consequently, no  $^{60}\text{Co}$  was detected in the samples. The  $^{137}\text{Cs}$  concentrations ranged from 882 to 1,400  $\mu\text{Ci/g}$ .

The composite was also analyzed for  $^{137}\text{Cs}$  and  $^{60}\text{Co}$  using GEA. However, this analysis was performed after a fusion digestion. The approximate analysis date was June 1998, and mean results of 783  $\mu\text{Ci/g}$  and 6.10  $\mu\text{Ci/g}$  were obtained for  $^{137}\text{Cs}$  and  $^{60}\text{Co}$ , respectively. Leach test results were performed directly and are described in Section B2.1.3.13.

**B2.1.3.9 Technetium-99.** Technetium-99 was analyzed by both liquid scintillation (individual auger, composite, and leach test samples) and ICP/MS (composite and leach test samples only). The liquid scintillation analysis on the individual auger samples was performed after an acid digestion. Procedure LA-438-101 was used in place of the procedure requested in the SAP (LA-438-112) because the SAP procedure was not appropriate due to the high concentrations of interfering analytes, which are difficult to remove from the filter. The analysis was rerun on the riser 9G samples several times because of difficulties in obtaining reproducible results. The digested sample and duplicate were reported as inhomogeneous because a slurry-like material would settle to the bottom of the bottles. The final analysis was performed under chemist supervision and the reported results were the highest of all the analyses performed. Only the data from the final analysis were included in Esch (1998) and used in deriving tank means. The previous sets of data for  $^{99}\text{Tc}$  were excluded because of concerns over proper stirring and subsampling of the material. The data for the unreported results are on file at the 222-S Laboratory and are available for review. Three out of four results from the riser 3A samples were below detection levels, while the riser 9G data ranged from 0.865 to 3.05  $\mu\text{Ci/g}$ .

The composite and leach test samples were analyzed for  $^{99}\text{Tc}$  using both liquid scintillation and ICP/MS. The composite sample was digested using fusion before analysis, while the leach test sample was analyzed directly. The composite results were 0.408  $\mu\text{Ci/g}$  from the liquid scintillation analysis and 0.646  $\mu\text{Ci/g}$  from the ICP/MS measurement. Leach test data are shown in Section B2.1.3.13.

An ICP/MS analysis was also run on an acid digested composite sample, although results from this analysis are not being included in this report. Results from the acid digested samples were two orders of magnitude lower than the results obtained by both ICP/MS and radiochemical methods after fusion digestion, suggesting that the acid digestion currently used in the 222-S-Laboratory may not be adequate for total dissolution of some Hanford Site tank wastes. Solids observed after the acid digestion further confirm this. These solids may include other metals in addition to  $^{99}\text{Tc}$  that were not dissolved during the digestion (Crawford 1998). The  $^{99}\text{Tc}$  acid digestion results for the composite are on file at the 222-S Laboratory.

**B2.1.3.10 Selenium-79.** Determinations of  $^{79}\text{Se}$  were performed by liquid scintillation counting on acid-digested subsamples of the individual auger samples. However, the  $^{79}\text{Se}$  data are considered suspect and should be used with caution. Liquid scintillation analysis generated similar count rates in the  $^{79}\text{Se}$  channel for both blanks and samples. For samples 97-AUG-001 and 97-AUG-002, both samples and blanks exhibited activity in the  $^{79}\text{Se}$  channel that was 2 to 2.5 times greater than the SAP requested detection limit. Esch (1998) attributes the activity observed in the  $^{79}\text{Se}$  channel to background counts caused by high levels of other radionuclides in the samples, especially  $^{137}\text{Cs}$ . Furthermore, the RPDs for three of the four auger samples were greater than the 20 percent level requested in the SAP. Because of these uncertainties in the  $^{79}\text{Se}$  data, a mean for  $^{79}\text{Se}$  activity was not derived. No further analyses were performed because reanalysis would have only produced the same results.

The composite was also analyzed by liquid scintillation on an acid-digested subsample. The concentration of  $^{79}\text{Se}$  in the composite was lower than the detection limit required by the HTI DQO and very close to the detection limit for the method. The blank results were at the same level as the reported sample results. The high blank results provide a measure of the uncertainty in the  $^{79}\text{Se}$  measurement. High concentrations of  $^{137}\text{Cs}$  in the sample can lead to positive interferences in the radiochemical analysis of low  $^{79}\text{Se}$  concentration because of inadequate  $^{137}\text{Cs}$  separation from  $^{79}\text{Se}$ . Based on these results and the individual auger sample results, the requirement for a  $^{79}\text{Se}$  analysis on the leach test sample was rescinded. Corroboration was obtained from the program and modelers who agreed that based on the  $^{79}\text{Se}$  content recorded in the AX tank farm flow sheets, the analyses were not necessary (Crawford 1998).

**B2.1.3.11 Alpha Energy Analysis for  $^{239/240}\text{Pu}$  and  $^{241}\text{Am}$ .** An alpha energy analysis (AEA) was only required for the individual auger samples. The analysis was performed on all of the auger samples after a fusion digestion, and on the riser 3A samples after acid digestion. Because the  $^{239/240}\text{Pu}$  data were being used in place of total alpha data, the results were compared against the total alpha notification limit of 34.2  $\mu\text{Ci/g}$ . None of the results exceeded this limit.

The riser 9G samples were analyzed twice on the original fusion digestion because of high relative percent differences (RPDs) between duplicate samples on the first analysis. The reanalysis still had RPDs greater than 20 percent, but they were accepted because of the apparent inhomogeneity of the sample. Only the second set of  $^{239/240}\text{Pu}$  results, which ranged from 4.67 to 8.61  $\mu\text{Ci/g}$ , were included in the data report (Esch 1998) and this tank characterization report.

Results for  $^{241}\text{Am}$  for the riser 9G samples varied between 14.7 and 26  $\mu\text{Ci/g}$ . Blank contamination was detected for the riser 3A samples during the  $^{241}\text{Am}$  analysis. Indications were that the contamination occurred during the sample analysis and not the sample digestion. Because the results were all less than the detection limit, no reanalysis was requested.

**B2.1.3.12 Fission Product Screening.** Screening was performed using ICP/MS to identify major fission products. The analysis was performed on acid-digested subsamples from the individual auger samples. Because no specific analytes were requested, the results are considered semi-quantitative. The ICP/MS instrument was directly calibrated for five masses (measured in atomic mass units, or AMUs). Table B2-7 lists the results for those five masses. Note that only the mass is reported, not the element. Without more extensive work, a definitive assignment is not possible. Attachment 3 of Esch (1998) provides further explanation of the fission product screening data and additional information including estimated concentrations of tentatively identified masses.

Table B2-7. Results of ICP/MS Screen.

Auger Sample	AMU 99	AMU 103	AMU 133	AMU 139	AMU 141
97-AUG-001	<12 ppb	<6 ppb	<37 ppb	<14 ppb	<10 ppb
97-AUG-002	<12 ppb	<6 ppb	<37 ppb	<14 ppb	<10 ppb
97-AUG-003	<1.2 ppm	$\approx 0.89$ ppm	<3.7 ppm	$\approx 3.6$ ppm	$\approx 3.4$ ppm
97-AUG-004	<1.2 ppm	<0.59 ppm	<3.7 ppm	$\approx 1.7$ ppm	$\approx 1.6$ ppm

**B2.1.3.13 Composite and Leach Test Results.** The composite and leach test data were obtained from *Tank 241-AX-104 Residual Solids Leach Test Results* (Crawford 1998).

The leachant was sampled four times during the leach test: after 1 day, 7 days, 30 days, and 90 days. The temperature and pH of the leachant were measured at the time of sampling. The temperature was also measured on a weekly basis. Throughout the study, the temperature ranged between 21 °C (70 °F) and 26.4 °C (79.5 °F), with an average temperature of 23 °C (74 °F). The pH changed from 6.62 (leachate solution with no solids added) to 6.32 after seven days. Within measurement uncertainty, no notable pH changes were observed after the first week of leaching. At the 30-day sampling, the pH of the unfiltered solution was 6.96, while the filtered sample had a pH of 6.37. The final pH (90-day sample) was 6.34.

Table B2-8 presents the composite and leach test results for the required analytes. Only sample means are reported; individual primary and duplicate results are available in Crawford (1998). Nondetected results (denoted by an "nd") are also available from Crawford (1998). Additionally,

data for all opportunistic analytes are presented in Crawford (1998). The 24 hour sample was obtained on the third day (at 48 hours from water contact and 24 hours after uninterrupted stirring). All other samples were obtained on the day required. All of the samples except the 30-day sample were analyzed within five days of sampling from the leach container. The 30-day sample was held up in process due to an unexpected laboratory outage that lasted over two weeks. It appears that the extended sample storage compromised the radionuclide and metals data, as lower values than expected were obtained. The lower values were likely a result of adsorption of metals to the storage container. The anions may also have been affected, although the nitrite trend appears consistent (Crawford 1998).

An in-depth interpretation of the composite and leach test analytical results is provided in Crawford (1998).

Table B2-8. Composite and Leach Test Results for the Required Analytes.<sup>1</sup> (2 sheets)

Analyte	Composite Concentration	24-Hour Sample	7-Day Sample	30-Day Sample	90-Day Sample
	µg/g	µg/mL	µg/mL	µg/mL	µg/mL
Ag	366	0.601	1.26	n/d	2.20
As	<229	<0.100	<0.100	n/d	n/d
Ba	1,560	5.98	5.54	n/d	n/d
Cd	1,440	0.855	1.20	n/d	3.68
Cr	494	n/d	n/d	n/d	n/d
Pb	7,310	n/d	n/d	n/d	n/d
Sb	<137	<0.020	<0.020	n/d	n/d
NO <sub>2</sub> <sup>-</sup>	15,700	456	1,720	2,150	7,940
NO <sub>3</sub> <sup>-</sup>	5.09E+05	22,400	50,100	23,900	41,300
Analyte	µCi/g	µCi/mL	µCi/mL	µCi/mL	µCi/mL
<sup>99</sup> Tc by ICP/MS	0.666	2.82E-04	2.82E-04	3.00E-04	7.20E-04
<sup>99</sup> Tc by liq. Scin.	0.408	4.49E-04	n/d	6.70E-05	9.23E-04
<sup>137</sup> Cs	783	9.99	19.8	10.9	6.09
<sup>60</sup> Co	6.10	n/d	n/d	n/d	n/d
<sup>79</sup> Se	3.87E-04	*	*	*	*

Table B2-8. Composite and Leach Test Results for the Required Analytes.<sup>1</sup> (2 sheets)

Analyte	Composite Concentration	24-Hour Sample	7-Day Sample	30-Day Sample	90-Day Sample
	μCi/g	μCi/mL	μCi/mL	μCi/mL	μCi/mL
Total alpha	41.5	n/d	n/d	n/d	n/d
<sup>90</sup> Sr by total beta <sup>2</sup>	46,600	2,240	3,500	1,150	537

## Notes:

liq. scin. = liquid scintillation

n/d = not detected

\* = not run

<sup>1</sup>Average of sample and duplicate values.<sup>2</sup>Strontium-90 values are calculated from the total beta analytical results by removing the <sup>137</sup>Cs and accounting for the <sup>90</sup>Y.**B2.2 VAPOR PHASE MEASUREMENTS**

Before the November 1997 auger sampling of tank 241-AX-104, a vapor phase measurement was taken for flammability issues. This measurement supported the safety screening DQO (Dukelow et al. 1995) and the organic solvents DQO (Meacham et al. 1997). A previous headspace vapor measurement had been made before the January 1997 vapor sampling event as discussed in Section B2.3. Both measurements were taken in the tank headspace 6.1 m (20 ft) below the top of riser 3A. All results were obtained in the field (that is, no gas sample was sent to a laboratory for analysis). For comparison purposes, the results from both events are shown in Table B2-9.

Table B2-9. Results of Headspace Measurements of Tank 241-AX-104.

Measurement	Result	
	November 1997	January 1997
TOC	0 ppm	0 ppm
LEL	0% of LEL	0% of LEL
Oxygen	20.9%	21%
Ammonia	0 ppm	0 ppm

### B2.3 DESCRIPTION OF 1997 VAPOR SAMPLING EVENT

On January 23, 1997, the headspace of tank 241-AX-104 was sampled using the in situ vapor sampling system. Sampling was performed in accordance with Revision 0 of *Vapor Sampling and Analysis Plan* (Buckley 1997) to support the Vapor Issue Resolution Program and the data needs of the *Data Quality Objective for Tank Hazardous Vapor Safety Screening* (Osborne and Buckley 1995). Three headspace sample types were collected: 1) SUMMA™ canister, 2) triple sorbent trap, and 3) sorbent tube train. SUMMA™ canister samples were analyzed for volatile organic compounds, total non-methane organic compounds, and permanent gases (specifically carbon dioxide, carbon monoxide, hydrogen, methane, and nitrous oxide). The triple sorbent traps were used to measure concentrations of volatile organic compounds. The sorbent tube train samples were used to determine the ammonia, nitric oxide, nitrogen dioxide, and water vapor concentrations. The samples were collected through riser 3A 11.6 m (38 ft) below the top of the adapter flange.

A thorough discussion of the sampling event is presented in *Tank Vapor Sampling and Analysis Data Package for Tank 241-AX-104, Sampled January 23, 1997* (Viswanath et al. 1997). Revised results for this sampling event were released in *Revised Data Tables for Tank Vapor Database on Tanks 241-A-106, 241-AX-104, and 241-TX-106* (Lockrem 1997). The data tables were revised because of incompatibilities in the data qualifiers between the laboratory and the tank vapor database; the actual data did not change between revisions.

Table B2-10 presents results for all detected analytes and the major nondetected analytes. No target analyte was detected at concentrations greater than its notification limit as specified in the vapor SAP. The major target organic analytes found in the headspace vapor SUMMA™ samples were acetone and n-pentane. For the triple sorbent trap samples, none of the target organic analytes were found at concentrations above the quantitation limit. Non-target organic compounds detected in the vapor samples are designated tentatively identified compounds. Two tentatively identified organic compounds were reported for the SUMMA™ samples, and four were reported for the triple sorbent trap samples (Viswanath et al. 1997). Of the nonorganic target analytes, only carbon dioxide and water vapor were above detection levels.



Table B2-10. Headspace Vapor Analytical Results for Tank 241-AX-104.<sup>1,2,3</sup> (2 sheets)

Analyte	Sample Device	Field Sample Number	Concentration	
			mg/m <sup>3</sup>	ppbv
Detected Organic Target Analytes				
Acetone	SUMMA™ canister	V7002-A04-014	0.036	14
		V7002-A04-014 DUP	0.039	15
n-pentane	SUMMA™ canister	V7002-A04-014	0.039	12
		V7002-A04-014 DUP	0.045	14
Tentatively Identified Compounds				
1,1,2-trichloro-1,2,2-trifluoroethane	SUMMA™ canister	V7002-A04-014	n/a	n/a
		V7002-A04-014 DUP	0.10	12
Benzaldehyde	SUMMA™ canister	V7002-A04-014	n/a	n/a
		V7002-A04-014 DUP	0.013	2.7
3,3,5-trimethylheptane	Triple sorbent trap	V7002-A12-032	0.23	36
3,8-dimethyldecane	Triple sorbent trap	V7002-A12-032	0.20	26
4-ethyl-2,2,6,6-tetramethylheptane	Triple sorbent trap	V7002-A12-032	0.37	45
3-methyl-5-propylnonane	Triple sorbent trap	V7002-A12-032	0.29	35
Carbon dioxide	SUMMA™ canister	V7002-A05-032	900	460 ppmv
Water vapor	Sorbent tube train	V7002-A07-T01	7,000	8,700 ppmv
		V7002-A08-T02	6,000	7,500 ppmv
		V7002-A09-T03	5,500	6,900 ppmv
		V7002-A10-T04	6,300	7,900 ppmv

Table B2-10. Headspace Vapor Analytical Results for Tank 241-AX-104.<sup>1,2,3</sup> (2 sheets)

Analyte	Sample Device	Field Sample Number	Concentration	
			mg/m <sup>3</sup>	ppbv
Major Nondetected Target Analytes				
Hydrogen	SUMMA™ canister	V7002-A05-032	<4.5	<50 ppmv
Nitrous oxide	SUMMA™ canister	V7002-A05-032	<98	<50 ppmv
Methane	SUMMA™ canister	V7002-A05-032	<36	<50 ppmv
Carbon monoxide	SUMMA™ canister	V7002-A05-032	<63	<50 ppmv
Non-methane total organic carbon	SUMMA™ canister	V7002-A05-032	<0.080	<150 ppbCv
Ammonia	Sorbent tube train	V7002-A07-T01; V7002-A08-T02; V7002-A09-T03; V7002-A10-T04	<11	<14 ppmv
Nitric oxide	Sorbent tube train	V7002-A07-T01; V7002-A08-T02; V7002-A09-T03; V7002-A10-T04	<5.5	<4.1 ppmv
Nitrogen dioxide	Sorbent tube train	V7002-A07-T01; V7002-A08-T02; V7002-A09-T03; V7002-A10-T04	<5.5	<2.7 ppmv

## Notes:

<sup>1</sup>Lockrem (1997)<sup>2</sup>Less-than values listed in the table are the Program Required Quantitation Limits for the target analytes, not the values of the method detection limits.<sup>3</sup>SUMMA is a trademark of Moletrics, Inc., Cleveland, Ohio.

Before attaching the in situ vapor sampling system to the tank, industrial hygiene field measurements were made to ensure worker protection. The tank vapor headspace was sampled at a depth of 6.1 m (20 ft) below the top of riser 3A. Measurements were made using a combustible gas indicator and an organic vapor meter (Viswanath et al. 1997). The results are presented in Table B2-9 in Section B2.2.

## B2.4 DESCRIPTION OF HISTORICAL SAMPLING EVENT

Four historical data sets exist for tank 241-AX-104. Two of those data sets are from sampling events that occurred before the tank was sluiced in 1977. Data obtained before the sluicing campaign are of limited value and are therefore not included in this TCR. Results from these two sampling events may be viewed in the tank 241-AX-104 file in the Tank Characterization and Safety Resource Center. The remaining two historical data sets are presented in the following subsections. Pre-1989 analytical data have not been validated and should be used with caution.

### B2.4.1 June 1978 Data Set

An analytical data set was reported in a June 1978 letter (Buckingham 1978). The data set was produced to study heat generation of the residual sludge in tank 241-AX-104. The letter report related that a sample of the residual sludge was analyzed for  $^{89/90}\text{Sr}$ ,  $^{137}\text{Cs}$ , and  $^{155}\text{Eu}$ . No information was provided regarding the sampling event, and it is unknown if the sample material was from an archived sample from the 1977 sampling or from a later sampling event. No mention of sample archival was made in the 1977 data report (Starr 1977).

No information regarding sample handling or analytical methods was provided in Buckingham (1978). Using data from the three reported radionuclides, Buckingham (1978) listed a calculated heat generation of 0.2316 W/L. The amount of sample received for analysis was very small, and there was a question as to how representative it was. To confirm the results, a sample of 241-AX-104 sludge that had been sluiced to tank 004-AR (in the AR vault) was analyzed. The tank 004-AR material was analyzed for  $^{89/90}\text{Sr}$  and  $^{137}\text{Cs}$ , and yielded a heat generation of 0.536 W/L. Buckingham (1978) indicated that the difference between the two analyses is enough to cast doubt on the representativeness of the 241-AX-104 sample.

The 1978 data set is displayed in Table B2-11. Both the 241-AX-104 data and the tank 004-AR data are presented.

Table B2-11. 1978 Data Set.<sup>1</sup> (2 sheets)

Analyte	Result	
	Ci/L	W/L
<b>Analysis of Residual 241-AX-104 Sludge</b>		
$^{89/90}\text{Sr}$	32.9	0.223
$^{137}\text{Cs}$	1.7	0.008
$^{155}\text{Eu}$	0.83	6E-04

Table B2-11. 1978 Data Set.<sup>1</sup> (2 sheets)

Analyte	Result	
	Ci/L	W/L
<b>Analysis of Tank 004-AR Sludge</b>		
<sup>89/90</sup> Sr	77.9	0.530
<sup>137</sup> Cs	1.26	0.006

Note:

<sup>1</sup>Pre-1989 data have not been validated and should be used with caution.

#### B2.4.2 September 1977 Sludge Sampling

A sample of the tank solids remaining after the first sluicing campaign was taken in September 1977. Although a second campaign was performed in 1978, the majority of the waste was removed during the first campaign. Therefore, with the exception of water content, the waste in the tank at the time of the 1977 sample should reasonably represent the current waste contents. The sampling method for the 1977 event was likely core sampling. The sample was received by the laboratory on September 15. The sample consisted of less than 10 mL of damp, dark sludge. Though malleable, it tended to retain its shape (did not flatten out when centrifuged at 1,500 gravities for several hours).

Analyses were performed directly on the sample and after water and fusion digestions. Measurements of water content, bulk density, particle density, and particle size distribution were made directly on the tank waste. For the water digestion, 4.8 mL of solids were washed with 15.7 mL of water. The wash solution was divided into two aliquots labeled JS20 and JS21, which were analyzed for selected species. The washed solids were then fused with potassium hydroxide and dissolved. Aliquots of the solution resulting from the fusion digestion, labeled JS22 and JS24, were subjected to comprehensive chemical analysis (Starr 1977).

Several days later, a second portion of the original sludge sample was fused with potassium hydroxide and dissolved with concentrated hydrochloric acid. An aliquot of the resulting solution (labeled JS25) was subjected to comprehensive chemical analysis. However, dissolution of this sample was incomplete. The precipitate was collected and washed, and the wash solution (labeled JS29) was brought to a pH of 8.3 using sodium borate in an effort to dissolve any acid-insoluble species that might be present. An aliquot of the wash was subjected to a battery of chemical analyses. The remaining precipitate was filtered and subjected to X-ray diffraction and elemental analysis. This qualitative analysis revealed the presence of several metals already

known to be present, predominantly potassium, iron, silicon, chromium and aluminum (Starr 1977). It was hypothesized that the fusion digestion did not go to completion because the potassium hydroxide did not completely cover the sample during fusion.

Table B2-12 presents the analytical results from the 1977 sampling event. Only one analytical result was reported for samples JS20 and JS21, implying that the results from the two samples were averaged before including in the data report (Starr 1977). The data for samples JS22 and JS24 were treated similarly. The author of the 1977 data transmittal letter believed that samples JS22/24 and JS25 best represented the chemical composition of the waste in tank 241-AX-104.

Table B2-12. Analytical Results from 1977 Sludge Sampling.<sup>1,2</sup> (2 sheets)

Analyte	Sample Number				Units
	JS20/21	JS22/24	JS25	JS29	
Metals					
Al	<0.013	1.21	3.72	<0.0208	M
Ba	n/r	0.022	0.020	<0.00428	M
Bi	n/r	n/r	n/r	0.00325	M
Ca	n/r	0.653	0.63	0.0317	M
Cd	n/r	n/r	0.015	0.00124	M
Cr	n/r	0.063	0.059	0.00221	M
Fe	n/r	4.67	5.95	0.081	M
Hg	n/r	n/r	1.44	0.00764	M
Mg	n/r	0.159	0.183	0.0442	M
Mn	n/r	0.091	0.078	<0.00246	M
Na	0.337	3.02	2.96	4.59	M
Ni	n/r	n/r	0.26	<0.00264	M
Si	n/r	1.80	3.17	0.324	M
Anions					
NO <sub>2</sub> <sup>-</sup>	0.083	n/r	n/r	---	M
NO <sub>3</sub> <sup>-</sup>	0.090	n/r	<1.14	0.290	M
OH <sup>-</sup>	15.6	---	---	---	M
SO <sub>4</sub> <sup>2-</sup>	0.010	0.125	<1.14	0.290	M
PO <sub>4</sub> <sup>3-</sup>	n/r	n/r	0.11	0.0102	M

Table B2-12. Analytical Results from 1977 Sludge Sampling.<sup>1,2</sup> (2 sheets)

Analyte	Sample Number				Units
	JS20/21	JS22/24	JS25	JS29	
Radionuclides					
<sup>60</sup> Co	---	0.11	---	---	Ci/L
<sup>137</sup> Cs	0.00857	1.94	1.60	0.00559	Ci/L
<sup>154</sup> Eu	---	0.26	---	---	Ci/L
<sup>155</sup> Eu	---	0.78	0.68	---	Ci/L
Pu	n/r	0.276	0.15	0.00556	g/L
<sup>106</sup> Rh	---	0.073	---	---	Ci/L
<sup>125</sup> Sb	---	0.72	0.53	0.0380	Ci/L
Sr	0.0133	105	69.7	0.0337	Ci/L
U	n/r	n/r	0.00425	0.00413	g/L
Total alpha	n/r	8.19E+09	4.89E+07	4,570 (μCi/L)	d/min/mL <sup>3</sup>

## Notes:

n/r = not reported

<sup>1</sup>Starr (1977)<sup>2</sup>Pre-1989 data have not been validated and should be used with caution.<sup>3</sup>d/min/mL = disintegrations per minute per milliliter

Table B2-13 presents the results for the physical property analyses. Although the results were listed within Starr (1977) on the data page for Samples JS22/24, the narrative indicated that these analyses were performed directly on sample material before any sample digestions.

Table B2-13. Physical Property Analytical Results for the 1977 Sample.<sup>1</sup> (2 sheets)

Physical Property	Result
Damp bulk density (as received)	1.8 g/mL
Particle density	1.7 g/mL
Water content	41%

Table B2-13. Physical Property Analytical Results for the 1977 Sample.<sup>1</sup> (2 sheets)

Physical Property	Result
Particle size distribution	Mass % of sample > <u>n</u> microns
	100.10% > 20 microns
	98.63% > 25 microns
	98.38% > 30 microns
	98.02% > 35 microns
	96.85% > 40 microns
	96.42% > 45 microns
	94.61% > 50 microns
	85.86% > 60 microns
	61.54% > 70 microns
	50.38% > 80 microns
	26.29% > 90 microns
	14.16% > 100 microns
	3.44% > 125 microns

Note:

<sup>1</sup>Pre-1989 data have not been validated and should be used with caution.**B2.5 DATA TABLES FOR THE NOVEMBER 1997 AUGER SAMPLES**

Table B2-14. Tank 241-AX-104 Analytical Results: Aluminum (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002301	Riser 3A	97-AUG-001	<202	<195	<199
S97T002302		97-AUG-002	<194	<202	<198
S97T002303	Riser 9G	97-AUG-003	50,500	47,000	48,800
S97T002304		97-AUG-004	57,500	56,600	57,100

Table B2-15. Tank 241-AX-104 Analytical Results: Antimony (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<243	<234	<239
S97T002302		97-AUG-002	<233	<243	<238
S97T002303	Riser 9G	97-AUG-003	<163	<167	<165
S97T002304		97-AUG-004	<333	<165	<249

Table B2-16. Tank 241-AX-104 Analytical Results: Arsenic (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<405	<391	<398
S97T002302		97-AUG-002	<388	<405	<397
S97T002303	Riser 9G	97-AUG-003	<272	<279	<276
S97T002304		97-AUG-004	<556	<276	<416

Table B2-17. Tank 241-AX-104 Analytical Results: Barium.

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<202	<195	<199
S97T002302		97-AUG-002	<194	<202	<198
S97T002303	Riser 9G	97-AUG-003	1,820	1,800	1,810
S97T002304		97-AUG-004	1,860	1,910	1,890



Table B2-18. Tank 241-AX-104 Analytical Results: Beryllium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<20.2	<19.5	<19.9
S97T002302		97-AUG-002	<19.4	<20.2	<19.8
S97T002303	Riser 9G	97-AUG-003	<13.6	<13.9	<13.8
S97T002304		97-AUG-004	<27.8	<13.8	<20.8

Table B2-19. Tank 241-AX-104 Analytical Results: Bismuth (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<405	<391	<398
S97T002302		97-AUG-002	<388	<405	<397
S97T002303	Riser 9G	97-AUG-003	<272	<279	<276
S97T002304		97-AUG-004	<556	<276	<416

Table B2-20. Tank 241-AX-104 Analytical Results: Boron (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<202	271	<237 <sup>QC,d</sup>
S97T002302		97-AUG-002	208	208	208
S97T002303	Riser 9G	97-AUG-003	<136	<139	<138
S97T002304		97-AUG-004	<278	<138	<208

Table B2-21. Tank 241-AX-104 Analytical Results: Cadmium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002301	Riser 3A	97-AUG-001	<20.2	<19.5	<19.9
S97T002302		97-AUG-002	<19.4	<20.2	<19.8
S97T002303	Riser 9G	97-AUG-003	1,810	1,720	1,770
S97T002304		97-AUG-004	1,470	1,510	1,490

Table B2-22. Tank 241-AX-104 Analytical Results: Calcium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002301	Riser 3A	97-AUG-001	1,250	2,700	1,980 <sup>QC:e</sup>
S97T002302		97-AUG-002	1,590	1,230	1,410 <sup>QC:e</sup>
S97T002303	Riser 9G	97-AUG-003	13,000	12,000	12,500
S97T002304		97-AUG-004	11,400	12,000	11,700

Table B2-23. Tank 241-AX-104 Analytical Results: Cerium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002301	Riser 3A	97-AUG-001	<405	<391	<398
S97T002302		97-AUG-002	<388	<405	<397
S97T002303	Riser 9G	97-AUG-003	2,810	2,780	2,800
S97T002304		97-AUG-004	2,800	2,880	2,840

Table B2-24. Tank 241-AX-104 Analytical Results: Chromium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	136	132	134
S97T002302		97-AUG-002	136	138	137
S97T002303	Riser 9G	97-AUG-003	586	548	567
S97T002304		97-AUG-004	601	573	587

Table B2-25. Tank 241-AX-104 Analytical Results: Cobalt (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<81	<78.1	<79.5
S97T002302		97-AUG-002	<77.5	<81	<79.3
S97T002303	Riser 9G	97-AUG-003	160	153	157
S97T002304		97-AUG-004	139	147	143

Table B2-26. Tank 241-AX-104 Analytical Results: Copper (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	54.8	53.8	54.3
S97T002302		97-AUG-002	63.8	60	61.9
S97T002303	Riser 9G	97-AUG-003	1,070	1,050	1,060
S97T002304		97-AUG-004	1,030	1,060	1,050

Table B2-27. Tank 241-AX-104 Analytical Results: Iron (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	4.93E+05	4.78E+05	4.86E+05 <sup>QC:d</sup>
S97T002302		97-AUG-002	4.69E+05	4.61E+05	4.65E+05
S97T002303	Riser 9G	97-AUG-003	2.69E+05	2.60E+05	2.65E+05
S97T002304		97-AUG-004	2.73E+05	2.81E+05	2.77E+05

Table B2-28. Tank 241-AX-104 Analytical Results: Lanthanum (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<202	<195	<199
S97T002302		97-AUG-002	<194	<202	<198
S97T002303	Riser 9G	97-AUG-003	1,460	1,460	1,460
S97T002304		97-AUG-004	1,450	1,510	1,480

Table B2-29. Tank 241-AX-104 Analytical Results: Lead (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	517	488	503
S97T002302		97-AUG-002	678	715	697
S97T002303	Riser 9G	97-AUG-003	8,960	8,480	8,720
S97T002304		97-AUG-004	9,740	9,900	9,820

Table B2-30. Tank 241-AX-104 Analytical Results: Lithium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002301	Riser 3A	97-AUG-001	<40.5	<39.1	<39.8
S97T002302		97-AUG-002	<38.8	<40.5	<39.6
S97T002303	Riser 9G	97-AUG-003	32.4	29.3	30.9
S97T002304		97-AUG-004	<55.6	27.7	<41.6

Table B2-31. Tank 241-AX-104 Analytical Results: Magnesium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002301	Riser 3A	97-AUG-001	2,450	2,520	2,490
S97T002302		97-AUG-002	1,610	1,650	1,630
S97T002303	Riser 9G	97-AUG-003	1,610	1,570	1,590
S97T002304		97-AUG-004	1,460	1,490	1,480

Table B2-32. Tank 241-AX-104 Analytical Results: Manganese (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002301	Riser 3A	97-AUG-001	3,080	3,000	3,040
S97T002302		97-AUG-002	2,770	2,770	2,770
S97T002303	Riser 9G	97-AUG-003	4,410	4,240	4,330
S97T002304		97-AUG-004	4,920	5,180	5,050

Table B2-33. Tank 241-AX-104 Analytical Results: Molybdenum (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<202	<195	<199
S97T002302		97-AUG-002	<194	<202	<198
S97T002303	Riser 9G	97-AUG-003	<136	<139	<138
S97T002304		97-AUG-004	<278	<138	<208

Table B2-34. Tank 241-AX-104 Analytical Results: Neodymium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<405	<391	<398
S97T002302		97-AUG-002	<388	<405	<397
S97T002303	Riser 9G	97-AUG-003	4,430	4,400	4,420
S97T002304		97-AUG-004	4,430	4,540	4,490

Table B2-35. Tank 241-AX-104 Analytical Results: Nickel (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	96.7	107	102
S97T002302		97-AUG-002	106	137	122 <sup>QC:e</sup>
S97T002303	Riser 9G	97-AUG-003	15,400	14,700	15,100
S97T002304		97-AUG-004	14,000	14,200	14,100

Table B2-36. Tank 241-AX-104 Analytical Results: Phosphorus (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<810	<781	<796 <sup>QC:d</sup>
S97T002302		97-AUG-002	<775	<810	<793
S97T002303	Riser 9G	97-AUG-003	700	<558	<629
S97T002304		97-AUG-004	<1110	900	<1,010

Table B2-37. Tank 241-AX-104 Analytical Results: Potassium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<2020	<1,950	<1,990 <sup>QC:d</sup>
S97T002302		97-AUG-002	<1940	<2,020	<1,980
S97T002303	Riser 9G	97-AUG-003	<1360	<1,390	<1,380
S97T002304		97-AUG-004	<2780	<1,380	<2,080

Table B2-38. Tank 241-AX-104 Analytical Results: Samarium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<405	<391	<398
S97T002302		97-AUG-002	<388	<405	<397
S97T002303	Riser 9G	97-AUG-003	1,040	980	1,010
S97T002304		97-AUG-004	965	1,010	988

Table B2-39. Tank 241-AX-104 Analytical Results: Selenium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<405	<391	<398 <sup>QC:c</sup>
S97T002302		97-AUG-002	<388	<405	<397
S97T002303	Riser 9G	97-AUG-003	<272	<279	<276
S97T002304		97-AUG-004	<556	<276	<416

Table B2-40. Tank 241-AX-104 Analytical Results: Silicon (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	528	694	611 <sup>QC:d,e</sup>
S97T002302		97-AUG-002	700	619	660
S97T002303	Riser 9G	97-AUG-003	1260	948	1,100 <sup>QC:e</sup>
S97T002304		97-AUG-004	955	310	633 <sup>QC:e</sup>

Table B2-41. Tank 241-AX-104 Analytical Results: Silver (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<40.5	<39.1	<39.8 <sup>QC:c</sup>
S97T002302		97-AUG-002	<38.8	<40.5	<39.6
S97T002303	Riser 9G	97-AUG-003	427	455	441
S97T002304		97-AUG-004	364	408	386



Table B2-42. Tank 241-AX-104 Analytical Results: Sodium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<405	397	<401 <sup>QC:d</sup>
S97T002302		97-AUG-002	388	<405	<397
S97T002303	Riser 9G	97-AUG-003	42,400	42,000	42,200
S97T002304		97-AUG-004	43,900	43,900	43,900

Table B2-43. Tank 241-AX-104 Analytical Results: Strontium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<40.5	<39.1	<39.8
S97T002302		97-AUG-002	<38.8	<40.5	<39.6
S97T002303	Riser 9G	97-AUG-003	967	950	959
S97T002304		97-AUG-004	932	961	947

Table B2-44. Tank 241-AX-104 Analytical Results: Sulfur (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<405	<391	<398 <sup>QC:d</sup>
S97T002302		97-AUG-002	<388	<405	<397
S97T002303	Riser 9G	97-AUG-003	1,430	1,350	1,390
S97T002304		97-AUG-004	1,780	1,760	1,770

Table B2-45. Tank 241-AX-104 Analytical Results: Thallium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<810	<781	<796 <sup>QC:c</sup>
S97T002302		97-AUG-002	<775	<810	<793
S97T002303	Riser 9G	97-AUG-003	<545	<558	<552
S97T002304		97-AUG-004	<1,110	<551	<831

Table B2-46. Tank 241-AX-104 Analytical Results: Titanium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<40.5	<39.1	<39.8
S97T002302		97-AUG-002	<38.8	<40.5	<39.6
S97T002303	Riser 9G	97-AUG-003	312	373	343
S97T002304		97-AUG-004	312	325	319

Table B2-47. Tank 241-AX-104 Analytical Results: Total Uranium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<2,020	<1,950	<1,990 <sup>QC:d</sup>
S97T002302		97-AUG-002	<1,940	<2,020	<1,980
S97T002303	Riser 9G	97-AUG-003	3,460	3,260	3,360
S97T002304		97-AUG-004	3,090	3,310	3,200

Table B2-48. Tank 241-AX-104 Analytical Results: Vanadium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<202	<195	<199
S97T002302		97-AUG-002	<194	<202	<198
S97T002303	Riser 9G	97-AUG-003	<136	<139	<138
S97T002304		97-AUG-004	<278	<138	<208

Table B2-49. Tank 241-AX-104 Analytical Results: Zinc (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	391	540	466 <sup>QC:e</sup>
S97T002302		97-AUG-002	405	350	378
S97T002303	Riser 9G	97-AUG-003	1,250	1,200	1,230
S97T002304		97-AUG-004	1,120	1,130	1,130

Table B2-50. Tank 241-AX-104 Analytical Results: Zirconium (ICP).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002301	Riser 3A	97-AUG-001	<40.5	<39.1	<39.8
S97T002302		97-AUG-002	<38.8	<40.5	<39.6
S97T002303	Riser 9G	97-AUG-003	3,540	3,560	3,550
S97T002304		97-AUG-004	4,310	4,400	4,360

Table B2-51. Tank 241-AX-104 Analytical Results: Bromide (IC).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002305	Riser 3A	97-AUG-001	<550	<531	<541
S97T002306		97-AUG-002	<300	<307	<304
S97T002307	Riser 9G	97-AUG-003	<566	<559	<562
S97T002308		97-AUG-004	<561	<552	<557

Table B2-52. Tank 241-AX-104 Analytical Results: Chloride (IC).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002305	Riser 3A	97-AUG-001	433	415	424
S97T002306		97-AUG-002	377	380	379
S97T002307	Riser 9G	97-AUG-003	412	386	399
S97T002308		97-AUG-004	245	209	227

Table B2-53. Tank 241-AX-104 Analytical Results: Fluoride (IC).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002305	Riser 3A	97-AUG-001	<52.8	<51	<51.9
S97T002306		97-AUG-002	<28.8	<29.5	<29.1
S97T002307	Riser 9G	97-AUG-003	108	101	104
S97T002308		97-AUG-004	96.7	98.4	97.6

Table B2-54. Tank 241-AX-104 Analytical Results: Nitrate (IC).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002305	Riser 3A	97-AUG-001	1.14E+05	1.13E+05	1.13E+05
S97T002306		97-AUG-002	1.04E+05	1.08E+05	1.06E+05
S97T002307	Riser 9G	97-AUG-003	61,300	65,500	63,400
S97T002308		97-AUG-004	26,500	29,400	28,000

Table B2-55. Tank 241-AX-104 Analytical Results: Nitrite (IC).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002305	Riser 3A	97-AUG-001	<475	<459	<467
S97T002306		97-AUG-002	<259	<266	<263
S97T002307	Riser 9G	97-AUG-003	2,370	2,380	2,380
S97T002308		97-AUG-004	2,060	2,170	2,110

Table B2-56. Tank 241-AX-104 Analytical Results: Oxalate (IC).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002305	Riser 3A	97-AUG-001	<462	<446	<454
S97T002306		97-AUG-002	<252	<258	<255
S97T002307	Riser 9G	97-AUG-003	2,760	<470	<1,620
S97T002308		97-AUG-004	<471	<464	<468

Table B2-57. Tank 241-AX-104 Analytical Results: Phosphate (IC).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002305	Riser 3A	97-AUG-001	<528	<510	<519
S97T002306		97-AUG-002	<288	<295	<292
S97T002307	Riser 9G	97-AUG-003	<543	<537	<540
S97T002308		97-AUG-004	<539	<530	<534

Table B2-58. Tank 241-AX-104 Analytical Results: Sulfate (IC).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
S97T002305	Riser 3A	97-AUG-001	1,080	898	987
S97T002306		97-AUG-002	396	373	384
S97T002307	Riser 9G	97-AUG-003	1,580	1,470	1,530
S97T002308		97-AUG-004	1,380	1,400	1,390

Table B2-59. Tank 241-AX-104 Analytical Results: Americium-241 (AEA).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
Solids: acid digest			μCi/g	μCi/g	μCi/g
S97T002301	Riser 3A	97-AUG-001	<0.00205	<0.00204	<0.00205
S97T002302		97-AUG-002	<0.00229	<0.00204	<0.00217
Solids: fusion			μCi/g	μCi/g	μCi/g
S97T002288	Riser 3A	97-AUG-001	0.00287	0.00245	0.00266 <sup>QC:f</sup>
S97T002289		97-AUG-002	0.00428	0.00382	0.00405 <sup>QC:f</sup>
S97T002290	Riser 9G	97-AUG-003	26	17	21.5 <sup>QC:e</sup>
S97T002291		97-AUG-004	14.7	17.8	16.3

Table B2-60. Tank 241-AX-104 Analytical Results: Cesium-137 (GEA).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$
S97T002301	Riser 3A	97-AUG-001	0.0753	0.0711	0.0732
S97T002302		97-AUG-002	0.184	0.18	0.182 <sup>QC:b</sup>
S97T002303	Riser 9G	97-AUG-003	1,400	1,330	1,370
S97T002304		97-AUG-004	889	882	885 <sup>QC:b</sup>

Table B2-61. Tank 241-AX-104 Analytical Results: Cobalt-60 (GEA).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			$\mu\text{Ci/g}$	$\mu\text{Ci/g}$	$\mu\text{Ci/g}$
S97T002301	Riser 3A	97-AUG-001	<0.0117	<0.0105	<0.0111
S97T002302		97-AUG-002	<0.00852	<0.00832	<0.00842
S97T002303	Riser 9G	97-AUG-003	<22.1	<25.3	<23.7 <sup>QC:b</sup>
S97T002304		97-AUG-004	<18.2	<9.01	<13.6

Table B2-62. Tank 241-AX-104 Analytical Results: Plutonium-239/240 (AEA).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
Solids: acid digest			μCi/g	μCi/g	μCi/g
S97T002301	Riser 3A	97-AUG-001	<0.00114	<0.00105	<0.0011
S97T002302		97-AUG-002	<0.0011	<0.00118	<0.00114
Solids: fusion			μCi/g	μCi/g	μCi/g
S97T002288	Riser 3A	97-AUG-001	<0.00252	<0.00233	<0.00243
S97T002289		97-AUG-002	<0.00228	<0.00231	<0.0023
S97T002290	Riser 9G	97-AUG-003	8.61	6.59	7.6 <sup>QC:e</sup>
S97T002291		97-AUG-004	4.67	6.76	5.71 <sup>QC:e</sup>

Table B2-63. Tank 241-AX-104 Analytical Results: Selenium-79 (Liquid Scintillation).<sup>1</sup>

Sample Number	Riser Number	Auger Sample	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>μCi/g</b>	<b>μCi/g</b>	<b>μCi/g</b>
S97T002301	Riser 3A	97-AUG-001	0.00509	0.00777	0.00643 <sup>QC:e,f</sup>
S97T002302		97-AUG-002	0.0107	0.00504	0.00787 <sup>QC:e,f</sup>
S97T002303	Riser 9G	97-AUG-003	<5.47E-04	5.82E-04	<5.65E-04 <sup>QC:f</sup>
S97T002304		97-AUG-004	0.00162	0.00117	0.00139 <sup>QC:e,f</sup>

Note:

<sup>1</sup>Data are considered suspect and should be used with caution. See Section B2.1.3.10.

Table B2-64. Tank 241-AX-104 Analytical Results: Strontium-89/90 (Beta Counting).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
Solids: acid digest			μCi/g	μCi/g	μCi/g
S97T002301	Riser 3A	97-AUG-001	1.14	1.22	1.18
S97T002302		97-AUG-002	2.35	2.45	2.4
Solids: fusion			μCi/g	μCi/g	μCi/g
S97T002288	Riser 3A	97-AUG-001	1.6	2.15	1.88 <sup>QC:e,f</sup>
S98T001174			3.86	4.44	4.15 <sup>QC:f</sup>
S97T002289		97-AUG-002	4.18	3.78	3.98 <sup>QC:f</sup>
S98T001175			53.7	151	102 <sup>QC:e</sup>
S97T002290	Riser 9G	97-AUG-003	53,600	40,900	47,300 <sup>QC:e</sup>
S98T001176			53,600	55,300	54,500 <sup>QC:b</sup>
S97T002291		97-AUG-004	26,300	39,000	32,700 <sup>QC:e</sup>
S98T001177			52,600	53,700	53,200 <sup>QC:b</sup>



Table B2-65. Tank 241-AX-104 Analytical Results: Technetium-99  
(Liquid Scintillation).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>μCi/g</b>	<b>μCi/g</b>	<b>μCi/g</b>
S97T002301	Riser 3A	97-AUG-001	<0.0172	<0.0165	<0.0169 <sup>QC:b</sup>
S97T002302		97-AUG-002	<0.0167	0.0181	<0.0174 <sup>QC:b</sup>
S97T002303	Riser 9G	97-AUG-003	1.17	3.05	2.11 <sup>QC:e</sup>
S97T002304		97-AUG-004	0.865	2.37	1.62 <sup>QC:e</sup>

Table B2-66. Tank 241-AX-104 Analytical Results: Total Alpha (Alpha Counting).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: acid digest</b>			<b>μCi/g</b>	<b>μCi/g</b>	<b>μCi/g</b>
S97T002301	Riser 3A	97-AUG-001	8.59E-04	0.00109	9.75E-04 <sup>QC:c,c</sup>
S97T002302		97-AUG-002	0.00124	0.00125	0.00125

Table B2-67. Tank 241-AX-104 Analytical Results: Percent Water.

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids</b>			<b>%</b>	<b>%</b>	<b>%</b>
S97T002280	Riser 3A	97-AUG-001	2.65	0.77	1.71 <sup>QC:e</sup>
S97T002281		97-AUG-002	12.3	12.7	12.4
S97T002282	Riser 9G	97-AUG-003	4.47	n/a	4.47
S97T002283		97-AUG-004	12.7	11.3	12

Table B2-68. Tank 241-AX-104 Analytical Results: Total Organic Carbon (Furnace Oxidation).

Sample Number	Riser Number	Auger Number	Result	Duplicate	Average
<b>Solids: water digest</b>			<b>µg/g</b>	<b>µg/g</b>	<b>µg/g</b>
S97T002305	Riser 3A	97-AUG-001	2,200	2,970	2,590 <sup>QC:e</sup>
S97T002306		97-AUG-002	2,200	2,930	2,570 <sup>QC:e</sup>
S97T002307	Riser 9G	97-AUG-003	<2,260	<2,240	<2,250
S97T002308		97-AUG-004	<2,240	<2,210	<2,230

### B3.0 ASSESSMENT OF CHARACTERIZATION RESULTS

This section discusses the overall quality and consistency of the current sampling results for tank 241-AX-104. This section also evaluates sampling and analysis factors that may impact data interpretation. These factors are used to assess overall data quality and consistency and to identify limitations in data use.

#### B3.1 FIELD OBSERVATIONS

No problems were noted during sampling. However, as described in the following paragraphs, the representativeness of the riser 3A samples to the majority of the tank solids is in question.

Substantial differences exist between the riser 9G samples and the riser 3A samples. The first observance of these differences occurred in the field. Chain-of-custody forms from the sampling event noted that high levels of contamination were present for the riser 9G samples, while no such mention was made for the riser 3A samples (Esch 1998). During laboratory operations, the radioactive material content of the riser 9G samples turned the clear glass storage containers black within three days. As shown in Table B3-1, a wide disparity exists in analytical results for many analytes. As expected, the riser 3A samples exhibited far less radioactivity than the riser 9G samples. Large differences were seen among key metal and anionic constituents also. In nearly all cases except iron and nitrate, the riser 3A samples contained significantly lower concentrations.

Table B3-1. Comparison of Data for Key Analytes.

Analyte	Riser 9G Mean	Riser 3A Mean	HDW Estimate
	µg/g or µCi/g	µg/g or µCi/g	µg/g or µCi/g
Aluminum	52,900	<199	0
Calcium	12,100	1,700	7,250
Iron	271,000	476,000	123,000
Nickel	14,600	112	2,540
Sodium	43,100	<399	68,900
Total uranium	3,280	<1,990	224
Nitrate	45,700	110,000	2.85E-12
<sup>241</sup> Am	18.9	0.00336	2.86
<sup>137</sup> Cs	1,130	0.128	463
<sup>239/240</sup> Pu	6.66	<0.0024	2.17
<sup>89/90</sup> Sr	46,900	28	12,000
<sup>99</sup> Tc	1.87	<0.0172	0.125

Table B3-1 also compares the means from each riser with the predicted concentrations based on process history (HDW estimate column). Although results may differ by an order of magnitude in some cases, most of the basic trends match between the tank 241-AX-104 HDW model estimates (based on PUREX high-level waste) and the riser 9G samples, especially for the radionuclides. The HDW model did not predict aluminum, which more closely matched the riser 3A result. The comparison was inconclusive for total uranium and nitrate.

Although Agnew et al. (1997) predicts only one waste type in the tank, the analytical results from riser 3A clearly indicate that a second "waste type" is present, or that the P2 waste under riser 3A has been altered in some way. A video of the waste surface, taken during a 1997 in-tank measurement campaign (Reich 1997), identified the presence of "debris" mounds under virtually every hardware item in the tank, including risers. During the tank's active service life, airlift circulators were used to inject cooling air into the waste. Injection of the air produced aerosolized waste that collected on all of the exposed tank surfaces. Over time, some of this collected waste has fallen off the riser surfaces and accumulated in a mound underneath each riser. Video images indicate that the debris has a significantly different texture and coloration from the other waste, and Reich (1997) remarked that the presence of the debris may bias a species inventory of any waste sample taken directly underneath a riser. Particular attention was paid to riser 3A in Reich (1997) because of the conflicting waste thickness data obtained during the 1997 in-tank measurement campaign. Temperature and radiation readings under the riser indicate a thin waste layer. However, magnetometer readings indicate a depth greater than 13 cm

(5 in.). Using video data, Reich (1997) estimated the debris mound under riser 3A to be 7.6 (3 in.)-thick. The data suggest that of the 13- to 15-cm (5- to 6-in.) waste depth under riser 3A, only approximately the bottom 5 cm (2 in.) would be representative of the entire waste profile. Because the stroke lengths for 97-AUG-001 and 97-AUG-002 were 7.6 (3 in.) and 9.8 (3 7/8 in.), respectively, the recovered material on the augers was likely composed of debris and therefore unrepresentative of the majority of the tank waste.

The in-tank measurements for riser 9G were more consistent. The magnetometer measurements yielded waste thickness readings between 4.3 and 12 cm (1.7 and 4.9 in.). The video data supported the magnetometer measurements, as a large waste mass is visible near the location of riser 9G. The video shows that riser 9G is on the edge of the mass, which would explain the different thickness readings. The temperature and radiation probe data also supported/confirmed the magnetometer waste thickness measurements (Reich 1997). Although an estimate of thickness for the debris mound under riser 9G was not made in Reich (1997), the mound would be expected to be smaller than the one under riser 3A. Riser 9G is only 15 cm (6 in.) in diameter and is located along the edge of the tank (see Appendix A), which would have reduced airflow. Only three airlift circulators are in the immediate vicinity of the riser, all on the right side. In contrast, riser 3A is 41 cm (16 in.) in diameter and is located near the center of the tank, almost in the middle of the two concentric rings of airlift circulators. Consequently, riser 3A would have been exposed to more aerosolized waste, and exposure would have come from all directions.

A portion of the debris under riser 3A is also believed to be tank corrosion products. Riser 3A has been used frequently in the past for gaining access to the tank waste. When risers are opened, debris or rust from the riser have been found to fall into the tank waste. Usually this is not a concern because of the large amount of waste and the small amount of rust. However, because tank 241-AX-104 does not contain much waste, a small amount of rust could potentially bias the analytical results. This was likely the case with the riser 3A samples, as demonstrated in the iron results. Auger samples 97-AUG-001 and 97-AUG-002 had mean iron results of 486,000 and 465,000 µg/g, respectively. These results are the highest iron values recorded for any tank on the Hanford Site. Except for one 202,000 µg/g result for tank 241-AW-106, the riser 3A iron results are nearly four times the results obtained on any other tank. The waste type in tank 241-AX-104 (PUREX high-level waste) is expected to have a high iron concentration, although the Agnew et al. (1997) estimate of 123,000 µg/g is still nearly four times below the riser 3A auger results. Riser 9G had seen limited use before the 1997 auger sampling. The riser 9G samples had means of 265,000 and 277,000 µg/g – more reasonable but also possibly showing a high bias as a result of some contamination by corrosion products.

Historical tank waste temperatures, which have averaged 36 °C (96 °F) since 1976, indicate that the waste should contain substantial amounts of radioactivity. The tank temperature data implies that a majority of the tank waste may be composed of the material sampled through riser 9G. If the waste were solely composed of the riser 9G material, the waste temperatures could be even higher; however, several of the parameters governing the thermal response of the tank (e.g., convective heat transfer) are not well defined. Section 2.5.2 presented a comparison of heat

loads based on radionuclide analytical data (using riser 9G results only) and tank waste temperatures. The analytical data-based heat load was approximately four times that derived from waste temperatures (Kummerer 1995).

Based on the available information, it is obvious that the material from risers 3A and 9G are substantially different, and that the riser 9G material is more closely related to the P2 waste type expected to be in the tank. Unfortunately, it is not known what fractions of waste the riser 3A samples and the riser 9G samples represent. For the purpose of deriving tank composition and inventory estimates, it was assumed that the fraction of waste represented by the riser 3A samples was minor compared to the P2 waste represented by the riser 9G samples; this assumption is principally based on the temperature data from the tank. Consequently, no data from the riser 3A samples were used in determining means and inventory estimates. Calculating means and inventories in this manner provides the most radiologically conservative estimates. However, omitting the riser 3A sample data may bias the estimates. Additional LDUA samples scheduled to be taken in the near future may determine the origin of the riser 3A samples and the fraction of the waste that they represent.

### **B3.2 QUALITY CONTROL ASSESSMENT**

The usual QC assessment includes an evaluation of the appropriate standard recoveries, spike recoveries, duplicate analyses, and blanks that are performed in conjunction with the chemical analyses. All pertinent QC tests were conducted on the 1997 auger samples, allowing a full assessment regarding the accuracy and precision of the data. The auger and LDUA SAPs (Schreiber 1998a and 1998b, respectively) established specific criteria for all analytes. Sample and duplicate pairs with one or more QC results outside the specified criteria were identified by footnotes in the data summary tables. Because the opportunistic analytes were not required by either SAP and therefore do not have defined QC parameters, a quality control assessment was not performed on the opportunistic data.

The standard and spike recovery results provide an estimate of analysis accuracy. If a standard or spike recovery is above or below the given criterion, the analytical results may be biased high or low, respectively. Nearly all standard recoveries were within the required limits. Two  $^{137}\text{Cs}$  standard recoveries, one  $^{90}\text{Sr}$  standard recovery, and one  $^{99}\text{Tc}$  standard recovery were slightly above the limit.

Matrix spike recoveries may have been affected for some analytes because of the incomplete transfer of sample material during the acid digestion. Spike recovery failures were noted for silicon, silver, iron, sodium, and uranium during the ICP analysis. The silicon failure may be attributed to "noise" near the detection limit, since most of the sample results were less than five times the detection limit. However, the subsamples had acid added to them before they were loaded out the hot cell, and sat in a vial for a longer time than usual before digestion. Leaching of silicon from the borosilicate glass may have occurred at this time as well as during the acid

digestion itself. The fact that not all glassware contains equal amounts of silicon may also account for the failures. The low recoveries for silver may have been the result of precipitation caused by an insufficient amount of hydrochloric acid used during digestion. The high spike recovery for sodium may have been caused by a combination of spectral interference from the high iron concentration, as well as matrix interference. Matrix interferences were also to blame for the high uranium recovery. The high spike recovery for iron indicates that the concentration in the sample was too high to perform a meaningful spike analysis. Post-digestion spike analyses were performed for silicon, silver, and uranium with acceptable results. A spike recovery for one total alpha analysis was slightly outside the target range. Because results were well below the action limit, no reruns were requested (Esch 1998).

The precision is estimated by the relative percent difference (RPD), which is defined as the absolute value of the difference between the primary and duplicate samples, divided by their mean, times 100. Most of the QC excursions were related to problems with precision. Precision was likely affected by the incomplete sample transfer during the acid dilution. Other reasons for precision problems included sample inhomogeneity (for  $^{239/240}\text{Pu}$ ,  $^{241}\text{Am}$ , and  $^{99}\text{Tc}$ ) and "noise" when results were at least two times below the detection limit (for silicon, nickel, and total organic carbon). Samples that had inhomogeneity problems were reanalyzed, and although RPDs did not improve, the results were accepted because of the inhomogeneity. One total alpha activity sample had a high RPD. The alpha activity was low for this sample, resulting in a high counting error. Because the results were well below the action limit, no reruns were requested. For  $^{90}\text{Sr}$ , three of the four samples had high RPDs on the first digest. The second digest had much lower RPDs, except for one sample. High RPDs were observed for three of the four  $^{79}\text{Se}$  samples. No further explanation regarding these samples was given in Esch (1998). Finally, precision problems were noted for the percent water data. One sample had a large RPD, while a second sample did not have a duplicate result because the beginning gross weight was less than the residual dry weight. Unfortunately, reruns could not be performed because of the need to conserve sample material for the leach tests (Esch 1998).

Contamination was detected in preparation blanks for several analytes, including silicon, sodium, nitrate, nitrite,  $^{90}\text{Sr}$ ,  $^{241}\text{Am}$  and  $^{79}\text{Se}$ . Because the silicon, sodium, nitrate, and nitrite contamination was minor, no reruns were requested for those samples. The amount of  $^{90}\text{Sr}$  in the preparation blank for the acid-digested subsamples was insignificant. The fusion digestion for  $^{90}\text{Sr}$  was prepared twice because of a high  $^{90}\text{Sr}$  concentration in the first blank. Results for the first set of fusion-digested samples (S97T002288 and -2289) from the riser 3A augers may not be meaningful because of the high blank contamination. However, because the results were actually lower than the blank, the sample and duplicate portions may not have been contaminated. The redigested samples still experienced minor blank contamination. Because the background level of strontium in the hot cell is high, it may be difficult to obtain a lower blank result. Contamination comparable to the reported concentrations was found in the  $^{241}\text{Am}$  blanks for the riser 3A samples. No contamination was found for the riser 9G samples, indicating the contamination occurred during the sample analysis instead of the digestion. Because the results for the riser 3A samples were below the requested detection limit, no reanalysis was performed. High blank values were found in all of the  $^{79}\text{Se}$  blanks. A scan of the blanks showed that there

was not an actual peak for  $^{79}\text{Se}$ , and that some of the counts were above the energy range where  $^{79}\text{Se}$  would be detected. This indicates that some of the activity detected was from radionuclides other than  $^{79}\text{Se}$ . Because of the relatively high concentrations of other radionuclides in these samples, further analysis would likely produce the same results. Therefore, no additional analysis was requested.

In summary, the vast majority of QC results were within the boundaries specified in the SAPs. In most cases, the discrepancies mentioned here and footnoted in the data summary tables should not impact data validity or use. As mentioned above, the  $^{90}\text{Sr}$  data from the first fusion digestion on the riser 3A samples should be used with caution. In addition, the water content data should be used carefully because of the difficulty in reproducing results. It should be noted that with respect to water content, visual observations supported the analytical results. Finally, the  $^{79}\text{Se}$  results are questionable.

A complete QC assessment, similar to that done on the individual auger samples, was performed in conjunction with the composite analyses and leach test. The discussion of the QC assessment provided below is limited to the required analytes.

Few QC problems were encountered. No problems with standard recoveries were noted in Crawford (1998). High spike results were obtained for nitrate and silver, while low spike recoveries were obtained for barium and cadmium. The high nitrate spike values may have resulted from using smaller spike concentrations in the presence of large concentrations of nitrate in the sample. Because the concentration of silver was small (less than  $400\text{ }\mu\text{g/g}$ ) and concentration differences were observed between duplicate samples (RPD of 23.5%), the high spike recovery for silver may have been the result of differences in the spiked and unspiked sample silver concentrations prior to sample spiking. For barium and cadmium, the spike concentrations used compared to the sample concentrations for both samples (approximately  $1,500\text{ }\mu\text{g/g}$ ) may have contributed to the low spike recovery. Precision problems were noted for silver and  $^{99}\text{Tc}$ . For  $^{99}\text{Tc}$ , the high RPDs from the liquid scintillation analysis were attributed to incomplete separation from interferents or variation in separation yields between methods. Low levels of blank contamination were noted for nitrate and  $^{79}\text{Se}$  (Crawford 1998).

To summarize, most of the QC results were within the required boundaries; the few QC excursions noted should not affect the usability of the data. Because of possible matrix effects (as shown by the spike and blank results), the nitrate results should be considered conservatively high.

The primary challenge in meeting the SAP requirements for the composite and leach tests was obtaining the stipulated detection limits. All of the metals (from the ICP/AES analysis) and anions (from the IC analysis) had detection limits above those required by the HTI DQO. For silver, barium, cadmium, chromium, lead, nitrate, and nitrite, this was not a concern because they were present in concentrations well above the detection limits. However, the antimony and arsenic concentrations were below detection limits. Because only confirmation of the presence

of a metal at or below the DQO-required detection limits was needed, these samples were evaluated by ICP/MS. The ICP/MS analysis revealed that antimony and arsenic were present in concentrations well below the DQO-required detection limits (Crawford 1998).

### **B3.3 DATA CONSISTENCY CHECKS**

Comparing different analytical methods is helpful in assessing the consistency and quality of the data. The auger sampling data set allowed a comparison of sulfur as analyzed by ICP to sulfate as analyzed by IC for the riser 9G samples. In addition, mass and charge balances were calculated to help assess the overall data consistency.

#### **B3.3.1 Comparison of Results from Different Analytical Methods**

The following data consistency check compares the results from two analytical methods. Agreement between the two methods strengthens the credibility of both results, while poor agreement may bring the reliability of the data into question. All analytical mean results were taken from the Section B2.5 tables.

The analytical sulfur mean result as determined by ICP was 1,580  $\mu\text{g/g}$ , which converts to 4,740  $\mu\text{g/g}$  of sulfate. This result did not compare well with the IC sulfate mean result of 1,460  $\mu\text{g/g}$ . The RPD between these two results is 106 percent, indicating that some of the sulfate may be bound in a water-insoluble compound.

#### **B3.3.2 Mass and Charge Balances**

The principle objective in performing mass and charge balances is to determine whether the measurements are consistent. In calculating the balances, the only analytes considered were those detected at a concentration of 1,000  $\mu\text{g/g}$  or greater as listed in Table B3-5 in Section B3.4.

Except for sodium, barium, and lead, all cations listed in Table B3-2 were assumed to be in their most common hydroxide form. Barium and lead were assumed to form compounds with sulfate. The concentrations of the assumed species were calculated stoichiometrically. Because precipitates are neutral species, all positive charge was attributed to the sodium cation. The anions listed in Table B3-3 were assumed to be present as sodium salts and were expected to balance the positive charge exhibited by the cations. The ICP sulfur data were used to generate the overall sulfate value, while the IC sulfate result was used to determine the water soluble portion. The remaining sulfate was assumed to form the compounds mentioned previously.



The concentrations of the cationic species in Table B3-2, the anionic species in Table B3-3, and the percent water were ultimately used to calculate the mass balance.

The mass balance was calculated from the formula below. The factor 0.0001 is the conversion factor from  $\mu\text{g/g}$  to weight percent.

$$\text{Mass balance} = \% \text{ water} + 0.0001 \times \{\text{total analyte concentration}\}$$

The total analyte concentration calculated from the above equation is 867,000  $\mu\text{g/g}$ . The mean weight percent water (obtained from the gravimetric mean reported in Table B3-5) is 8.27 percent or 82,700  $\mu\text{g/g}$ . The mass balance resulting from adding the percent water to the total analyte concentration is 95.0 percent (shown in the "Subtotal" row of Table B3-3). As described below, hydroxide is assumed to balance the initial net positive charge calculated for the waste. The mass of hydroxide needed to balance the charge is 17,900  $\mu\text{g/g}$ . This value is in good agreement with the best-basis hydroxide estimate of 19,600  $\mu\text{g/g}$ . Adding the 17,900  $\mu\text{g/g}$  to the total analyte concentration subtotal produces an overall analyte concentration of 968,000  $\mu\text{g/g}$ , or 96.8 percent. Ideally, a mass balance should equal 100 percent. A value less than 100 percent indicates that the full concentration of one or more tank constituents was not measured. One possible explanation for this is incomplete digestion of the sample material. It is known that some aluminum compounds are resistant to digestion by acid. Because the ICP subsamples for tank 241-AX-104 were prepared after an acid digestion, it is possible that not all of the aluminum was measured. Also, carbonate was not measured. The HDW model predicts that 10,900  $\mu\text{g/g}$  should be present based on process history.

The following equations demonstrate the derivation of total cations and total anions; the charge balance is the ratio of these two values.

$$\text{Total cations } (\mu\text{eq/g}) = [\text{Na}^+]/23.0 = 1,870 \mu\text{eq/g}$$

$$\text{Total anions } (\mu\text{eq/g}) = [\text{NO}_3^-]/62.0 + [\text{NO}_2^-]/46.0 + [\text{SO}_4^{2-}]/96.0 = 816 \mu\text{eq/g}$$

The charge balance obtained by dividing the sum of the positive charge by the sum of the negative charge was 2.29, indicating that an anionic species was missing. This missing species was assumed to be hydroxide, which was not measured on the auger samples. To balance the charge, 17,900  $\mu\text{g/g}$  of hydroxide were added to the calculation.

In summary, the above calculations reveal that at least one waste constituent may not have been fully measured during the sample analysis. Also, it is estimated that hydroxide exists as the second most abundant anion in the waste.

Table B3-2. Cation Mass and Charge Data for Solids.

Analyte	Concentration ( $\mu\text{g/g}$ )	Assumed Species	Concentration of Assumed Species ( $\mu\text{g/g}$ )	Charge ( $\mu\text{eq/g}$ )
Al	52,900	$\text{Al}(\text{OH})_3$	153,000	0
Ba	1,850	$\text{BaSO}_4$	3,140	0
Ca	12,100	$\text{Ca}(\text{OH})_2$	22,400	0
Cd	1,630	$\text{Cd}(\text{OH})_2$	2,120	0
Ce	2,820	$\text{Ce}(\text{OH})_3$	3,850	0
Cu	1,050	$\text{Cu}(\text{OH})_2$	1,610	0
Fe	271,000	$\text{Fe}(\text{OH})_3$	518,000	0
La	1,470	$\text{La}(\text{OH})_3$	2,010	0
Mg	1,530	$\text{Mg}(\text{OH})_2$	3,670	0
Mn	4,690	$\text{Mn}(\text{OH})_4$	10,500	0
Na	43,100	$\text{Na}^+$	43,100	1,870
Nd	4,450	$\text{Nd}(\text{OH})_3$	6,020	0
Ni	14,600	$\text{Ni}(\text{OH})_2$	23,100	0
Pb	9,270	$\text{PbSO}_4$	6,290	0
		$\text{Pb}(\text{OH})_2$	5,790	0
U	3,280	$\text{U}(\text{OH})_6$	4,690	0
Zn	1,180	$\text{Zn}(\text{OH})_2$	1,790	0
Zr	3,950	$\text{Zr}(\text{OH})_4$	6,890	0
Totals			818,000	1,870

Table B3-3. Anion Mass and Charge Data for Solids.

Analyte	Concentration (µg/g)	Assumed Species	Concentration of Assumed Species (µg/g)	Charge (µeq/g)
NO <sub>3</sub> <sup>-</sup>	45,700	NO <sub>3</sub> <sup>-</sup>	45,700	737
NO <sub>2</sub> <sup>-</sup>	2,240	NO <sub>2</sub> <sup>-</sup>	2,240	48.7
SO <sub>4</sub> <sup>2-</sup>	4,740 <sup>1</sup>	SO <sub>4</sub> <sup>2-</sup>	1,460 <sup>2</sup>	30.4
		BaSO <sub>4</sub>	3,140 <sup>3</sup>	0
		PbSO <sub>4</sub>	6,290 <sup>3</sup>	0
Totals			49,400	816

## Notes:

<sup>1</sup>Value is converted from the ICP sulfur data.<sup>2</sup>Soluble portion derived from the IC sulfate data.<sup>3</sup>The masses for these species are already included in Table B3-2.

Table B3-4. Mass and Charge Balance Totals.

Totals	Concentrations ( $\mu\text{g/g}$ )	Charge ( $\mu\text{eq/g}$ )
Total from Table B3-2 (cations)	818,000	1,870
Total from Table B3-3 (anions)	49,400	-816
Water percent	82,700	---
Subtotal	950,000	1,054
Amount of hydroxide needed to balance charge	17,900	-1,054
Grand totals	968,000	0

**B3.4 MEAN CONCENTRATIONS AND CONFIDENCE INTERVALS**

A nested analysis of variance (ANOVA) model was fit to the riser 9G sample data only; the riser 3A samples were deemed not representative of the majority of the tank waste and were not included in the ANOVA model. Mean values and 95 percent confidence intervals on the mean were determined from the ANOVA. Four variance components were used in the calculations. The variance components represent concentration differences between risers, segments, laboratory samples, and analytical replicates. The model is:

$$Y_{ijk} = \mu + R_i + S_{ij} + L_{ijk} + A_{ijkm},$$

$$i=1,2,\dots,a; j=1,2,\dots,b_i; k=1,2,\dots,c_{ij}; m=1,2,\dots,n_{ijk}$$

where

$Y_{ijkm}$  = concentration from the  $m^{\text{th}}$  analytical result of the  $k^{\text{th}}$  sample of the  $j^{\text{th}}$  segment of the  $i^{\text{th}}$  riser

$\mu$  = the mean

$R_i$  = the effect of the  $i^{\text{th}}$  riser

$S_{ij}$  = the effect of the  $j^{\text{th}}$  segment from the  $i^{\text{th}}$  riser

$L_{ijk}$  = the effect of the  $k^{\text{th}}$  sample from the  $j^{\text{th}}$  segment of the  $i^{\text{th}}$  riser

$A_{ijkm}$  = the analytical error

$a$  = the number of risers

$b_i$  = the number of segments from the  $i^{\text{th}}$  riser

$c_{ij}$  = the number of samples from the  $j^{\text{th}}$  segment of the  $i^{\text{th}}$  riser

$n_{ijk}$  = the number of analytical results from the  $ijk^{\text{th}}$  sample.

The variables  $R_i$ ,  $S_{ij}$ , and  $L_{ijk}$  are random effects. These variables, as well as  $A_{ijkm}$ , are assumed to be uncorrelated and normally distributed with means zero and variances  $\sigma^2(R)$ ,  $\sigma^2(S)$ ,  $\sigma^2(L)$  and  $\sigma^2(A)$ , respectively.

The restricted maximum likelihood method (REML) was used to estimate the mean concentration and standard deviation of the mean for all analytes that had 50 percent or more of their reported values greater than the detection limit. The mean value and standard deviation of the mean were used to calculate the 95 percent confidence intervals. The following table gives the mean, degrees of freedom, and confidence interval for each constituent.

Some analytes had results that were below the detection limit. In these cases, the value of the detection limit was used for non-detected results. Using the detection limit value to estimate the

analyte concentration in the non-detect samples may cause the calculated mean to be biased high. For analytes with a majority of results below the detection limit, a simple average is all that is reported.

The lower and upper limits, LL(95%) and UL(95%), of a two-sided 95 percent confidence interval on the mean were calculated using the following equation:

$$\begin{aligned} \text{LL}(95\%) &= \hat{\mu} - t_{(df, 0.025)} \times \hat{\sigma}(\hat{\mu}), \\ \text{UL}(95\%) &= \hat{\mu} + t_{(df, 0.025)} \times \hat{\sigma}(\hat{\mu}). \end{aligned}$$

In this equation,  $\hat{\mu}$  is the REML estimate of the mean concentration,  $\hat{\sigma}(\hat{\mu})$  is the REML estimate of the standard deviation of the mean, and  $t_{(df, 0.025)}$  is the quantile from Student's  $t$  distribution with  $df$  degrees of freedom. The degrees of freedom equal the number of laboratory samples minus one. In cases where the lower limit of the confidence interval was negative, it is reported as zero. Note that statistics were not calculated for  $^{79}\text{Se}$  because the data are considered suspect (see Section B2.1.3.10).

Table B3-5. Tank 241-AX-104 95 Percent Two-Sided Confidence Interval for the Mean Concentration for Solid Sample Data.<sup>1</sup> (Reference Date – October 23, 1998) (3 sheets)

Analyte	Method	Mean	df	LL	UL	Units
Aluminum	ICP:A	5.29E+04	1	1.69E+02	1.06E+05	µg/g
Americium-241	AEA:F	1.89E+01	1	0.00E+00	5.22E+01	µCi/g
Antimony <sup>2</sup>	ICP:A	<2.07E+02	n/a	n/a	n/a	µg/g
Arsenic <sup>2</sup>	ICP:A	<3.46E+02	n/a	n/a	n/a	µg/g
Barium	ICP:A	1.85E+03	1	1.37E+03	2.32E+03	µg/g
Beryllium <sup>2</sup>	ICP:A	<1.73E+01	n/a	n/a	n/a	µg/g
Bismuth <sup>2</sup>	ICP:A	<3.46E+02	n/a	n/a	n/a	µg/g
Boron <sup>2</sup>	ICP:A	<1.73E+02	n/a	n/a	n/a	µg/g
Bromide <sup>2</sup>	IC:W	<5.60E+02	n/a	n/a	n/a	µg/g
Cadmium	ICP:A	1.63E+03	1	0.00E+00	3.37E+03	µg/g
Calcium	ICP:A	1.21E+04	1	7.02E+03	1.72E+04	µg/g
Cerium	ICP:A	2.82E+03	1	2.53E+03	3.10E+03	µg/g
Cesium-137	GEA:A	1.13E+03	1	0.00E+00	4.18E+03	µCi/g
Chloride	IC:W	3.13E+02	1	0.00E+00	1.41E+03	µg/g
Chromium	ICP:A	5.77E+02	1	4.34E+02	7.20E+02	µg/g
Cobalt	ICP:A	1.50E+02	1	6.40E+01	2.36E+02	µg/g

Table B3-5. Tank 241-AX-104 95 Percent Two-Sided Confidence Interval for the Mean Concentration for Solid Sample Data.<sup>1</sup> (Reference Date – October 23, 1998) (3 sheets)

Analyte	Method	Mean	df	LL	UL	Units
Cobalt-60 <sup>2</sup>	GEA:A	<1.86E+01	n/a	n/a	n/a	μCi/g
Copper	ICP:A	1.05E+03	1	9.44E+02	1.16E+03	μg/g
Fluoride	IC:W	1.01E+02	1	5.76E+01	1.44E+02	μg/g
Iron	ICP:A	2.71E+05	1	1.91E+05	3.50E+05	μg/g
Lanthanum	ICP:A	1.47E+03	1	1.30E+03	1.64E+03	μg/g
Lead	ICP:A	9.27E+03	1	2.28E+03	1.63E+04	μg/g
Lithium <sup>2</sup>	ICP:A	3.63E+01	1	0.00E+00	1.19E+02	μg/g
Magnesium	ICP:A	1.53E+03	1	8.02E+02	2.26E+03	μg/g
Manganese	ICP:A	4.69E+03	1	8.15E+01	9.29E+03	μg/g
Molybdenum <sup>2</sup>	ICP:A	<1.73E+02	n/a	n/a	n/a	μg/g
Neodymium	ICP:A	4.45E+03	1	4.01E+03	4.89E+03	μg/g
Nickel	ICP:A	1.46E+04	1	8.54E+03	2.06E+04	μg/g
Nitrate	IC:W	4.57E+04	1	0.00E+00	2.71E+05	μg/g
Nitrite	IC:W	2.24E+03	1	5.73E+02	3.91E+03	μg/g
Oxalate <sup>2</sup>	IC:W	<1.04E+03	n/a	n/a	n/a	μg/g
Percent water	Percent solids	8.27E+00	1	0.00E+00	5.61E+01	%
Phosphate <sup>2</sup>	IC:W	<5.37E+02	n/a	n/a	n/a	μg/g
Phosphorus <sup>2</sup>	ICP:A	8.17E+02	1	0.00E+00	3.21E+03	μg/g
Plutonium-239/240	PU239/240:F	6.66E+00	1	0.00E+00	1.86E+01	μCi/g
Potassium <sup>2</sup>	ICP:A	<1.73E+03	n/a	n/a	n/a	μg/g
Samarium	ICP:A	9.99E+02	1	7.87E+02	1.21E+03	μg/g
Selenium <sup>2</sup>	ICP:A	<3.46E+02	n/a	n/a	n/a	μg/g
Silicon	ICP:A	8.68E+02	1	0.00E+00	3.86E+03	μg/g
Silver	ICP:A	4.14E+02	1	6.41E+01	7.63E+02	μg/g
Sodium	ICP:A	4.31E+04	1	3.22E+04	5.39E+04	μg/g
Strontium	ICP:A	9.53E+02	1	8.55E+02	1.05E+03	μg/g
Strontium-89/90	Sr:F	4.69E+04	3	3.10E+04	6.28E+04	μCi/g
Sulfate	IC:W	1.46E+03	1	5.95E+02	2.32E+03	μg/g
Sulfur	ICP:A	1.58E+03	1	0.00E+00	3.99E+03	μg/g
Technetium-99	Tc99:A	1.86E+00	1	0.00E+00	8.37E+00	μCi/g
Thallium <sup>2</sup>	ICP:A	<6.91E+02	n/a	n/a	n/a	μg/g
Titanium	ICP:A	3.31E+02	1	1.46E+02	5.15E+02	μg/g
Total organic	Furnace	<2.24E+03	n/a	n/a	n/a	μg/g

Table B3-5. Tank 241-AX-104 95 Percent Two-Sided Confidence Interval for the Mean Concentration for Solid Sample Data.<sup>1</sup> (Reference Date – October 23, 1998) (3 sheets)

Analyte	Method	Mean	df	LL	UL	Units
carbon <sup>2</sup>	oxidation:W					
Uranium	ICP:A	3.28E+03	1	2.26E+03	4.30E+03	µg/g
Vanadium <sup>2</sup>	ICP:A	<1.73E+02	n/a	n/a	n/a	µg/g
Zinc	ICP:A	1.18E+03	1	5.40E+02	1.81E+03	µg/g
Zirconium	ICP:A	3.95E+03	1	0.00E+00	9.07E+03	µg/g

## Notes:

<sup>1</sup>Riser 9G sample data only were included in the ANOVA model.<sup>2</sup>Less than values were used in the mean calculation.**B4.0 APPENDIX B REFERENCES**

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**APPENDIX C**

**STATISTICAL ANALYSIS FOR ISSUE RESOLUTION**

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## APPENDIX C

### STATISTICAL ANALYSIS FOR ISSUE RESOLUTION

Appendix C documents the results of the analyses and statistical and numerical manipulations required by the DQOs applicable to tank 241-AX-104. The analyses required for tank 241-AX-104 are reported as follows:

- **Section C1.0:** Statistical analysis and numerical manipulations supporting the safety screening DQO (Dukelow et al. 1995).
- **Section C2.0:** Appendix C references.

#### C1.0 STATISTICS FOR THE SAFETY SCREENING DATA QUALITY OBJECTIVE

The safety screening DQO (Dukelow et al. 1995) defines decision limits in terms of one-sided 95 percent confidence intervals. The safety screening DQO limits are 34.2  $\mu\text{Ci/g}$  for total alpha and 480 J/g for DSC. Total alpha analyses were only performed for the riser 3A samples. As directed by Schreiber (1998), total alpha analyses were deleted from the analytical suite for the riser 9G samples because  $^{239/240}\text{Pu}$  was already being analyzed. Therefore,  $^{239/240}\text{Pu}$  results were used in this safety screening comparison for the riser 9G samples. Confidence intervals calculated for the total alpha activity and  $^{239/240}\text{Pu}$  means from each laboratory sample are presented in Table C1-1. Schreiber (1998) also replaced the DSC analysis with a TOC analysis by furnace oxidation. However, only the riser 3A samples had TOC data above detection limits (Esch 1998); therefore, confidence intervals are calculated for the riser 3A data only. The decision limit for TOC concentration is 45,000  $\mu\text{g C/g}$  (Adams 1998) on a dry-weight basis.

The upper limit (UL) of a one-sided 95 percent confidence interval on the mean is

$$\hat{\mu} + t_{(df,0.05)} \hat{\sigma}_{\mu}$$

In this equation,  $\hat{\mu}$  is the arithmetic mean of the data,  $\hat{\sigma}_{\mu}$  is the estimate of the standard deviation of the mean, and  $t_{(df,0.05)}$  is the quantile from Student's t distribution with  $df$  degrees of freedom. The degrees of freedom equal the number of samples minus one. For sample numbers with at least one value above the detection limit, the UL of a 95 percent confidence interval is

given in Table C1-1 for total alpha/ $^{239/240}\text{Pu}$  and in Table C1-2 for TOC. Each confidence interval can be used to make the following statements. For total alpha activity and  $^{239/240}\text{Pu}$ , if the upper limit is less than 34.2  $\mu\text{Ci/g}$ , then one would reject the null hypothesis that the alpha or  $^{239/240}\text{Pu}$  is greater than or equal to 34.2  $\mu\text{Ci/g}$  at the 0.05 level of significance. For TOC, if the upper limit is less than 45,000  $\mu\text{g C/g}$ , then one would reject the null hypothesis that the TOC is greater than or equal to 45,000  $\mu\text{g C/g}$  at the 0.05 level of significance.

The riser 3A total alpha activity results showed little activity in the samples, as evidenced by the low 95 percent confidence interval ULs (0.00170 and 0.00128  $\mu\text{Ci/g}$ ). For the  $^{239/240}\text{Pu}$  results from the riser 9G samples, the UL closest to the threshold was 14.0  $\mu\text{Ci/g}$  for auger sample 97-AUG-003. This is nearly two and a half times below the limit of 34.2  $\mu\text{Ci/g}$ .

Table C1-1. 95 Percent Upper Confidence Limits for Total Alpha Activity and  $^{239/240}\text{Pu}$ .

Lab Sample ID	Description	$\hat{\mu}$	$df$	UL	Units
S97T002301	97-AUG-001	9.75 E-04	1	0.00170	$\mu\text{Ci/g}$
S97T002302	97-AUG-002	0.00125	1	0.00128	$\mu\text{Ci/g}$
S97T002290	97-AUG-003	7.60	1	14.0	$\mu\text{Ci/g}$
S95T002291	97-AUG-004	5.72	1	12.3	$\mu\text{Ci/g}$

The TOC sample results were converted to a dry-weight basis before calculation of the confidence intervals. The largest 95 percent confidence interval UL for the riser 3A TOC data was 5,550  $\mu\text{g C/g}$ , over eight times below the 45,000  $\mu\text{g C/g}$  limit. As mentioned previously, all TOC results for the riser 9G samples were below detection levels.

Table C1-2. 95 Percent Upper Confidence Limits for Total Organic Carbon.

Lab Sample ID	Description	$\hat{\mu}$	$df$	UL	Units
S97T002305	97-AUG-001	2,630	1	5,090	$\mu\text{g C/g}$
S97T002306	97-AUG-002	2,930	1	5,550	$\mu\text{g C/g}$

## C2.0 APPENDIX C REFERENCES

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**APPENDIX D**

**EVALUATION TO ESTABLISH BEST-BASIS INVENTORY  
FOR SINGLE-SHELL TANK 241-AX-104**



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**APPENDIX D****EVALUATION TO ESTABLISH BEST-BASIS INVENTORY  
FOR SINGLE-SHELL TANK 241-AX-104**

An effort is underway to provide waste inventory estimates that will serve as standard characterization source terms for the various waste management activities (Hodgson and LeClair 1996) at the Hanford Site. As part of this effort, an evaluation of available information for single-shell tank 241-AX-104 was performed, and a best-basis inventory was established. This work, detailed in the following sections, follows the methodology established by the standard inventory task.

**D1.0 CHEMICAL INFORMATION SOURCES**

Tank 241-AX-104 was most recently sampled in November 1997 to determine the composition of residual waste in the tank. Four different auger samples were taken from different locations in the tank. The samples were mechanically homogenized, subsampled, and analyzed for selected anions, cations, and radionuclides. No density measurements were obtained. These data are reported in Esch (1998), and the samples from riser 9G will be considered the primary data source for information about the composition of the waste in tank 241-AX-104. The samples from riser 3A do not appear to be representative of waste that originated from fuel processing. Instead, these samples may be contaminated debris resulting from tank corrosion or dried aerosol waste. The amount of waste represented by the riser 3A samples is assumed to be insignificant compared to the actual fuel-processing waste in the tank and will therefore not be included in the derivation of inventories for this tank. Omission of the riser 3A data will provide radiologically conservative inventories.

Substantial uncertainty exists with regard to the volume of waste still remaining in this tank. Reich (1997) has the most comprehensive information regarding the remaining waste volume in tank 241-AX-104. The current volume range for tank 241-AX-104 is 18.9 to 28.4 kL (5 to 7.5 kgal). The estimated volume used for deriving inventories in this effort was 28.4 kL (7.5 kgal).

Other sources of information regarding this tank include a previous core sample taken in September 1977 to determine the composition of the residual sludge remaining after the 1977 sluicing campaign (Starr 1977). Comprehensive chemical and solubility information was taken from this sample. These data were the basis for the previous best-basis estimates (LMHC 1998),

and will be used when the current data are not sufficient. The density measurement of the waste at that time, 1.80 g/mL, will be used in calculating inventories.

Samples taken prior to 1977 are of limited value because they reflect the composition of the waste before the sluicing campaign, and data from these efforts will not be used in developing best-basis estimates for tank 241-AX-104. The HDW model (Agnew et al. 1997a) provides tank content estimates in terms of component concentrations and inventories, using process knowledge and assumptions about the physical and chemical behavior of the wastes in the tanks.

A comprehensive waste history of this tank is provided in Agnew et al. (1997b).

Tank 241-AX-104 was primarily used as a PUREX high-level process waste receiver from the third quarter of 1966 until the second quarter of 1969, followed by the receipt and transfer of various supernatants from or to other tanks. The remaining supernatant was pumped out for cesium recovery during the third quarter of 1976, while most of the sludge (196.2 kL [52 kgal]) was sluiced for strontium recovery during the second and third quarters of 1977. A second sluicing campaign was conducted during the first quarter of 1978 to remove most of the residual sludge from this tank (Rodenhizer 1987). Tank 241-AX-104 was declared an assumed leaker in 1977, with an estimated leakage volume of 30.2 kL (8 kgal). A solids volume reevaluation was made in May 1978 (Everly 1978), and interim stabilization was completed in August 1981 (Hanlon 1998).

## D2.0 COMPARISON OF COMPONENT INVENTORY VALUES

In January 1993, the average sludge depth was estimated to be 6.98 cm (2.75 in.) in this tank (Swaney 1993). This depth is slightly higher than that derived from the tank farm surveillance estimate (6.48 cm [2.55 in.]), using 26.5 kL (7 kgal) as a basis (Hanlon 1998). Based on the Swaney (1993) estimate, tank 241-AX-104 contains about 28.7 kL (7.58 kgal) of waste (in the 22.9-m [75-ft]-diameter, flat bottom tank). Further analysis done by Reich (1997) provides a volume range of 18.9 to 28.4 kL (5 to 7.5 kgal), because video surveillance of the tank interior indicates that the waste is irregularly distributed. All of this waste consists of dried, highly friable sludge. For purposes of this analysis, the best-basis inventory will be developed from the high-end estimate derived by Reich (1997), 28.4 kL [7.5 kgal] of sludge.

Table D2-1 provides a summary of the washed sludge analyses from the 1977 sludge sample and tank inventory estimates based on the volume and density of the sludge (28.4 kL [7.5 kgal] and 1.8 kg/L, respectively), together with the wash solution components. Because this sample was obtained after the 1977 sluicing campaign, and formed the basis for the previous best-basis inventory estimates, the estimates derived from this sample will be compared against estimates derived from the information in Esch (1998) and the HDW model (Agnew et al. 1997a). (The

chemical species are reported without change designation in accordance with the best-basis inventory convention.)

Table D2-1. Inventory Estimates for Selected Nonradioactive Components in Tank 241-AX-104. (2 sheets)

Component	1977 Sample Data Tank Inventory Estimate <sup>1</sup> (kg)	1997 Sample Data Tank Inventory Estimate <sup>2</sup> (kg)	HDW Total Tank Inventory <sup>3</sup> (kg)
Al	1,890	2,700	0
Ba	81.9	94.4	nr
Bi	nr	<17.7	0
Ca	729	619	258
Cd	47.9	83.2	nr
Cl	nr	16.0	19.8
Cr	90.1	29.5	8.96
F	nr	5.16	0
Fe	8,420	13,800	4,380
La	nr	75.1	0
K	nr	<88.4	4.75
Mg	118	78.3	nr
Mn	132	240	0
Na	1,950	2,200	2,450
Ni	433	745	90.4
NO <sub>2</sub>	108 <sup>4</sup>	115	715
NO <sub>3</sub>	158 <sup>4</sup>	2,340	1.01E-13
OH <sub>TOTAL</sub>	7,530 <sup>4</sup>	19,600 <sup>5</sup>	4,120
Pb	nr	474	0
PO <sub>4</sub>	297	128	0
Si	1,980	44.4	1,130
SO <sub>4</sub>	341	242	328
Sr	nr	48.7	0
U <sub>TOTAL</sub>	0.121	168	7.99
Zr	nr	202	0

Table D2-1. Inventory Estimates for Selected Nonradioactive Components in Tank 241-AX-104. (2 sheets)

<b>Component</b>	<b>1977 Sample Data Tank Inventory Estimate<sup>1</sup> (kg)</b>	<b>1997 Sample Data Tank Inventory Estimate<sup>2</sup> (kg)</b>	<b>HDW Total Tank Inventory<sup>3</sup> (kg)</b>
TOC	nr	<114	0
H <sub>2</sub> O (wt%)	41	8.27	61.5

## Notes:

nr = not reported

<sup>1</sup>LMHC (1998). Data from Starr (1977), except for Na. Tank inventory based on 28.4 kL (7.5 kgal) of sludge with an average density of 1.80 kg/L; mean concentration based on the average of the JS22/24 and JS25 samples from Table B2-12.

<sup>2</sup>Source data from Esch (1998). Tank inventory based on 28.4 kL (7.5 kgal) of sludge with an average density of 1.80 kg/L; mean concentrations taken from Table B3-5.

<sup>3</sup>Agnew et al. (1997a) Tank inventory based on 26.5 kL (7.0 kgal) of sludge with an average density of 1.34 kg/L.

<sup>4</sup>Inventory based on the concentration for samples JS20/21 (water digest).

<sup>5</sup>OH<sub>TOTAL</sub> was not directly determined but was calculated from the charge balance.

Table D2-2 provides a comparison of the mean sludge radionuclide tank inventory estimates based on the 1977 grab sample, 1997 auger sample, and the HDW model. Radionuclide results in Table D2-2 have been decayed to January 1, 1994.

Table D2-2. Inventory Estimates for Radioactive Components in Tank 241-AX-104. (Decayed to January 1, 1994) (2 sheets)

<b>Component</b>	<b>1977 Sample Data Tank Inventory Estimate<sup>1</sup> (Ci)</b>	<b>1997 Sample Data Tank Inventory Estimate<sup>2</sup> (Ci)</b>	<b>HDW Total Tank Inventory<sup>3</sup> (Ci)</b>
<sup>60</sup> Co	334	<1,610	0.868
<sup>79</sup> Se	nr	0.05	2.95

Table D2-2. Inventory Estimates for Radioactive Components in Tank 241-AX-104.  
(Decayed to January 1, 1994) (2 sheets)

<b>Component</b>	<b>1977 Sample Data Tank Inventory Estimate<sup>1</sup> (Ci)</b>	<b>1997 Sample Data Tank Inventory Estimate<sup>2</sup> (Ci)</b>	<b>HDW Total Tank Inventory<sup>3</sup> (Ci)</b>
<sup>90</sup> Sr	1.64E+06	2.64E+06	4.28E+05
<sup>90</sup> Y	1.64E+06	2.64E+06	4.29E+05
<sup>99</sup> Tc	nr	95.3	4.47
<sup>125</sup> Sb	248	nr	3.90
<sup>137</sup> Cs	34,000	63,300	16,500
<sup>137m</sup> Ba	32,200	59,900	15,600
<sup>154</sup> Eu	1,870	nr	194
<sup>155</sup> Eu	1,700	nr	250
<sup>239/240</sup> Pu	375	340	77.0
<sup>241</sup> Am	nr	972	102

Notes:

nr = not reported

<sup>1</sup>LMHC (1998). Data from Starr (1977). Tank inventory based on 28.4 kL (7.5 kgal) of sludge with an average density of 1.80 kg/L. Concentration data taken from Table B2-12 (assumed analysis date of October 1977); mean concentrations based on the average of the JS22/24 and JS25 samples.

<sup>2</sup>Source data from Esch (1998). Tank inventory based on 28.4 kL (7.5 kgal) of sludge with an average density of 1.80 kg/L. Concentration data taken from Table B3-5 (assumed analysis date of March 1998).

<sup>3</sup>Agnew et al. (1997a). Tank inventory based on 26.5 kL (7.0 kgal) of sludge with an average density of 1.34 kg/L.

### D3.0 COMPONENT INVENTORY EVALUATION

Sample-based estimates developed from analytical data and HDW model estimates from Los Alamos National Laboratory (Agnew et al. 1997a) are both potentially useful for estimating component inventories in the tank. The HDW model is mainly based on process production records and waste transaction records for each tank. Primary wastes are process wastes initially added to tank 241-AX-104 from the processing plant, while secondary wastes are transferred into

the tank from another tank. A review of these records shows that tank 241-AX-104 received the following wastes (Agnew et al. 1997b):

- 7,521 kL (1,987 kgal) of primary PUREX high-level (P2) waste, most of which was later sluiced to B Plant for strontium recovery during the 1977 sluicing campaign
- 897 kL (237 kgal) of PUREX organic wash (OWW3) waste, all of which was later sluiced to B Plant for strontium recovery during the 1977 sluicing campaign because the OWW3 sludge layer was added to the top of the P2 layer
- 185 kL (49 kgal) of high-level B Plant (B) waste added in 1968 and 1969, all of which was later sluiced to B Plant for strontium recovery during the 1977 sluicing campaign
- 485 kL (128 kgal) of PUREX low-level (PL) waste, all of which was later sluiced to B Plant for strontium recovery during the 1977 sluicing campaign
- 34 kL (9 kgal) of B Plant AR vault sludge added in early 1976, all of which was later sluiced to B Plant for strontium recovery during the 1977 sluicing campaign
- Various supernatant transfers from tanks 241-A-102, 241-A-104, and 241-AX-103 from 1973 to 1976.

The HDW model (Agnew et al. 1997a) assumes that 26.5 kL (7 kgal) of PUREX high-level (P2) sludge were left in this tank after the 1977 and 1978 sluicing campaigns. The sludge inventory estimates derived from this model are consistent with the tank farm surveillance data for this tank (26.5 kL [7 kgal] of sludge) (Hanlon 1998).

### **D3.1 WASTE TYPES**

Of the five types of waste added to tank 241-AX-104, the most important from a volume perspective are P2, OWW3, and PL. However, only P2 waste remains in the tank in any substantial quantity because of the sluicing campaigns that were undertaken in 1977 and 1978.

### **D3.2 SUMMARY**

The 1997 auger sample data were used to generate inventory estimates for most of the chemical components in the tank 241-AX-104 waste. Data from the 1977 sample were used to supplement

the more recent data where analytes were not measured. Where neither sample provided an estimate, Agnew et al. (1997a) was used.

#### **D4.0 DEFINE THE BEST-BASIS AND ESTABLISH COMPONENT INVENTORIES**

Chemical and radionuclide inventory estimates are generally derived from one of three sources of information: 1) sample analysis and sample-derived inventory estimates, 2) component inventories predicted by the HDW model based on process knowledge and historical tank transfer information, or 3) a tank-specific process estimate based on process flowsheets, reactor fuel data, essential materials records, or comparable sludge layers and sample information from other tanks.

An effort is currently underway to provide waste inventory estimates that will serve as the standard characterization data for various waste management activities. As part of this effort, a survey and analysis of various sources of information relating to the chemical and radionuclide component inventories in tank 241-AX-104 was performed, including the following:

1. Data from auger samples obtained in 1997 (Esch 1998).
2. Data from a solids sample obtained in 1977 after the first sluicing campaign (Starr 1977).
3. Component inventory estimates provided by the HDW model (Agnew et al. 1997a).

Based on this analysis, a best-basis inventory was developed. The 1997 auger samples were used to generate estimates for most of the chemical and radionuclide components in this waste. This waste mostly consists of PUREX high-level waste from Al-clad fuel. The best-basis inventory for tank 241-AX-104, based on a waste volume of 28.4 kL (7.5 kgal) is presented in Tables D4-1 and D4-2. The inventory values reported in Tables D4-1 and D4-2 are subject to change. Refer to the Tank Characterization Database for the most current inventory values.

Once the best-basis inventories were determined, the hydroxide inventory was calculated by performing a charge balance with the valences of other analytes. This charge balance approach is consistent with that used by Agnew et al. (1997a).

Best-basis tank inventory values are derived for 46 key radionuclides (as defined in Section 3.1 of Kupfer et al. 1998), all decayed to a common report date of January 1, 1994. Often, waste sample analyses have only reported  $^{90}\text{Sr}$ ,  $^{137}\text{Cs}$ ,  $^{239/240}\text{Pu}$ , and total uranium (or total beta and total alpha), while other key radionuclides such as  $^{60}\text{Co}$ ,  $^{99}\text{Tc}$ ,  $^{129}\text{I}$ ,  $^{154}\text{Eu}$ ,  $^{155}\text{Eu}$ , and  $^{241}\text{Am}$ , etc., have been infrequently reported. For this reason it has been necessary to derive most of the 46 key



radionuclides by computer models. These models estimate radionuclide activity in batches of reactor fuel, account for the split of radionuclides to various separations plant waste streams, and track their movement with tank waste transactions. These computer models are described in Kupfer et al. (1998), Section 6.1 and in Watrous and Wootan (1997). Model-generated values for radionuclides in any of the 177 Hanford Site tanks are reported in the HDW Rev. 4 model results (Agnew et al. 1997a). The best-basis value for any one analyte may be either a model result or a sample or engineering assessment-based result if available. For a discussion of typical error between model-derived values and sample-derived values, see Kupfer et al. (1998), Section 6.1.10.

Table D4-1. Best-Basis Inventory Estimates for Nonradioactive Components in Tank 241-AX-104. (Effective December 10, 1998) (2 sheets)

Analyte	Total Inventory (kg)	Basis (S, M, E or C) <sup>1</sup>	Comment
Al	2,700	S	Starr (1977) = 1,890
Bi	0	E	No process history of Bi
Ca	619	S	Starr (1977) = 729
Cl	16.0	S	
TIC as CO <sub>3</sub>	557	M/E	HDW model concentration value scaled to 28.4 kL (7.5 kgal)
Cr	29.5	S	Starr (1977) = 90.1
F	5.16	S	
Fe	13,900	S	Starr (1977) = 8,420
Hg	0	E	Simpson (1998)
K	88.4	S/E	Upper bounding estimate
La	75.1	S	
Mn	240	S	Starr (1977) = 132
Na	2,200	S	Starr (1977) = 1,950
Ni	433	S	Starr (1977)
NO <sub>2</sub>	115	S	Starr (1977) = 108
NO <sub>3</sub>	2,340	S	Starr (1977) = 158
OH <sub>TOTAL</sub>	19,600	C	Based on charge balance
Pb	474	S	
PO <sub>4</sub>	128	S	Based on ICP. Starr (1977) = 297
Si	44.4	S	QC problems with 1997 value. Starr (1977) = 1,980

Table D4-1. Best-Basis Inventory Estimates for Nonradioactive Components in Tank 241-AX-104. (Effective December 10, 1998) (2 sheets)

Analyte	Total Inventory (kg)	Basis (S, M, E or C) <sup>1</sup>	Comment
SO <sub>4</sub>	242	S	Based on ICP. Starr (1977) = 341
Sr	48.7	S	
TOC	114	S/E	Upper bounding estimate
U <sub>TOTAL</sub>	168	S	Starr (1977) = 0.121
Zr	202	S	

Note:

<sup>1</sup>S = sample-based, M = HDW model-based, (Agnew et al. 1997a), E = engineering assessment-based, and C = calculated by charge balance; includes oxides as hydroxides, not including CO<sub>3</sub>, NO<sub>2</sub>, NO<sub>3</sub>, PO<sub>4</sub>, SO<sub>4</sub>, and SiO<sub>3</sub>.

Table D4-2. Best-Basis Inventory Estimates for Radioactive Components in Tank 241-AX-104, Decayed to January 1, 1994. (Effective December 10, 1998) (3 sheets)

Analyte	Total inventory (Ci)	Basis (S, M, or E) <sup>1</sup>	Comment
<sup>3</sup> H	3.38	M	
<sup>14</sup> C	0.63	M	
<sup>59</sup> Ni	3.12	M	
<sup>60</sup> Co	334	S	Starr (1977)
<sup>63</sup> Ni	313	M	
<sup>79</sup> Se	0.0500	S	<sup>79</sup> Se suffered from QC failures
<sup>90</sup> Sr	2.64 E+06	S	
<sup>90</sup> Y	2.64 E+06	S	Referenced to <sup>90</sup> Sr
<sup>93m</sup> Nb	10.2	M	
<sup>93</sup> Zr	13.6	M	
<sup>99</sup> Tc	95.3	S	
<sup>106</sup> Ru	0.0101	M	
<sup>113m</sup> Cd	58.2	M	
<sup>125</sup> Sb	3.90	M	
<sup>126</sup> Sn	4.61	M	
<sup>129</sup> I	0.00864	M	
<sup>134</sup> Cs	0.207	M	

Table D4-2. Best-Basis Inventory Estimates for Radioactive Components in Tank 241-AX-104, Decayed to January 1, 1994. (Effective December 10, 1998) (3 sheets)

Analyte	Total inventory (Ci)	Basis (S, M, or E) <sup>1</sup>	Comment
<sup>137</sup> Cs	63,300	S	
<sup>137m</sup> Ba	59,900	S	Referenced to <sup>137</sup> Cs
<sup>151</sup> Sm	11,000	M	
<sup>152</sup> Eu	3.36	M	
<sup>154</sup> Eu	1,870	S	Starr (1977)
<sup>155</sup> Eu	1,700	S	Starr (1977)
<sup>226</sup> Ra	1.96 E-04	M	
<sup>227</sup> Ac	0.00106	M	
<sup>228</sup> Ra	1.77 E-09	M	
<sup>229</sup> Th	2.77 E-07	M	
<sup>231</sup> Pa	0.00238	M	
<sup>232</sup> Th	1.60 E-10	M	
<sup>232</sup> U	4.67E-06	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>233</sup> U	1.10E-07	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>234</sup> U	0.0576	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>235</sup> U	0.00240	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>236</sup> U	0.00157	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>237</sup> Np	0.00954	M	
<sup>238</sup> Pu	11.0	S/M	Based on <sup>239</sup> Pu and HDW isotopic distribution
<sup>238</sup> U	0.0561	S/M	Based on U <sub>TOTAL</sub> and HDW isotopic distribution
<sup>239</sup> Pu	286	S/M	Based on <sup>239/240</sup> Pu and HDW isotopic distribution
<sup>240</sup> Pu	54.5	S/M	Based on <sup>239/240</sup> Pu and HDW isotopic distribution
<sup>241</sup> Am	972	S	

Table D4-2. Best-Basis Inventory Estimates for Radioactive Components in Tank 241-AX-104, Decayed to January 1, 1994. (Effective December 10, 1998) (3 sheets)

Analyte	Total inventory (Ci)	Basis (S, M, or E) <sup>1</sup>	Comment
<sup>241</sup> Pu	785	S/M	Based on <sup>239</sup> Pu and HDW isotopic distribution
<sup>242</sup> Cm	0.888	S/M	Based on <sup>241</sup> Am and HDW isotopic distribution
<sup>242</sup> Pu	0.00454	S/M	Based on <sup>239</sup> Pu and HDW isotopic distribution
<sup>243</sup> Am	0.0298	S/M	Based on <sup>241</sup> Am and HDW isotopic distribution
<sup>243</sup> Cm	0.0682	S/M	Based on <sup>241</sup> Am and HDW isotopic distribution
<sup>244</sup> Cm	2.10	S/M	Based on <sup>241</sup> Am and HDW isotopic distribution

Note:

<sup>1</sup>Sample-based, M =HDW model-based (Agnew et al. 1997a), and E = engineering assessment-based.

## D5.0 APPENDIX D REFERENCES

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**APPENDIX E**

**BIBLIOGRAPHY FOR TANK 241-AX-104**



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**APPENDIX E****BIBLIOGRAPHY FOR TANK 241-AX-104**

Appendix E is a bibliography that supports the characterization of tank 241-AX-104. This bibliography represents an in-depth literature search of all known information sources that provide sampling, analysis, surveillance, modeling information, and processing occurrences associated with tank 241-AX-104 and its respective waste types.

The references in this bibliography are separated into three categories containing references broken down into subgroups. These categories and their subgroups are listed below.

**I. NON-ANALYTICAL DATA**

- Ia. Models/Waste Type Inventories/Campaign Information
- Ib. Fill History/Waste Transfer Records
- Ic. Surveillance/Tank Configuration
- Id. Sample Planning/Tank Prioritization
- Ie. Data Quality Objectives/Customers of Characterization Data

**II. ANALYTICAL DATA - SAMPLING OF TANK WASTE AND WASTE TYPES**

- IIa. Sampling of Tank 241-AX-104
- IIb. Sampling of PUREX High-Level Waste

**III. COMBINED ANALYTICAL/NON-ANALYTICAL DATA**

- IIIa. Inventories Using Both Campaign and Analytical Information
- IIIb. Compendium of Existing Physical and Chemical Documented Data Sources

This bibliography is broken down into the appropriate sections of material with an annotation at the end of each reference describing the information source. Most information listed below is available in the Lockheed Martin Hanford Corporation Tank Characterization and Safety Resource Center.

## **I. NON-ANALYTICAL DATA**

### **Ia. Models/Waste Type Inventories/Campaign Information**

Anderson, J. D., 1990, *A History of the 200 Area Tank Farms*, WHC-MR-0132, Westinghouse Hanford Company, Richland, Washington.

- Contains single-shell tank fill history and primary campaign and waste information to 1981.

Jungfleisch, F. M., and B. C. Simpson, 1993, *Preliminary Estimation of the Waste Inventories in Hanford Tanks Through 1980*, WHC-SD-WM-TI-057, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

- A model based on process knowledge and radioactive decay estimations using ORIGEN for different compositions of process waste streams assembled for total, solution, and solids compositions per tank. Assumptions about waste/waste types and solubility parameters and constraints are also given.

### **Ib. Fill History/Waste Transfer Records**

Agnew, S. F., R. A. Corbin, T. B. Duran, K. A. Jurgensen, T. P. Ortiz, and B. L. Young, 1997, *Waste Status and Transaction Record Summary (WSTRS) Rev. 4*, LA-UR-97-311, Rev. 0, Los Alamos National Laboratory, Los Alamos, New Mexico.

- Contains spreadsheets showing all available data on tank additions and transfers.

Anderson, J. D., 1990, *A History of the 200 Area Tank Farms*, WHC-MR-0132, Westinghouse Hanford Company, Richland, Washington.

- Contains single-shell tank fill history and primary campaign and waste information to 1981.

Rodenhizer, D. G., 1987, *Hanford Waste Tank Sluicing History*, SD-WM-TI-302, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

- Contains a history of sluicing operations on Hanford Site waste tanks.

**Ic. Surveillance/Tank Configuration**

Alstad, A. T., 1993, *Riser Configuration Document for Single-Shell Waste Tanks*, WHC-SD-RE-TI-053, Rev. 9, Westinghouse Hanford Company, Richland, Washington.

- Shows tank riser locations in relation to a tank aerial view and describes risers and their contents.

Anantatmula, R. P., 1997, *Evaluation of Recent Data from Single-Shell Tank 241-AX-104*, (internal memorandum 74711-97-RPA-070 to R. A. Dodd, September 11), Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Analyzes magnetometer and radiation level data from tank 241-AX-104 in order to explain discrepancies between risers 3A and 9G.

Bailey, J. W., 1978, *Tank Status Update*, (letter 60412-78-0434 to Distribution, October 2), Rockwell Hanford Operations, Richland, Washington.

- Declares the official status change from Inactive to Inactive-Primary Stabilized for tank 241-AX-104. Gives a supernatant liquid estimate of zero gallons.

Bath, S. S., 1977, *Heat Study on Tank 104-AX With and Without Air Cooling*, (letter 0256 to J. W. Bailey, December 16), Rockwell Hanford Operations, Richland, Washington.

- Contains results from a heat study done to determine the feasibility of pumping the liquid out of the tank and cooling it with air.

Lipnicki, J., 1997, *Waste Tank Risers Available for Sampling*, HNF-SD-RE-TI-710, Rev. 4, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Assesses riser locations for each tank; however, not all tanks are included or completed. The risers believed to be available for sampling are also included.

Reich, F. R., 1997, *241-AX-104 Residual Waste Volume Estimate*, HNF-SD-HTI-ER-001, Rev. 0, SGN Eurisys Services Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Provides an estimate of the residual waste volume in tank 241-AX-104 based on in-tank measurements and archived volume data.

Tran, T. T., 1993, *Thermocouple Status Single-Shell & Double-Shell Waste Tanks*, WHC-SD-WM-TI-553, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

- Contains riser and thermocouple information for Hanford Site waste tanks.

Welty, R. K., 1988, *Waste Storage Tank Status and Leak Detection Criteria*, SD-WM-TI-356, Westinghouse Hanford Company, Richland, Washington.

- Presents liquid level, drywall, and leak detection pit surveillance data along with a tank status summary.

#### **Id. Sample Planning/Tank Prioritization**

Adams, M. R., T. M. Brown, J. W. Hunt, and L. J. Fergestrom, 1998, *Fiscal Year 1999 Waste Information Requirements Document*, HNF-2884, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains Tri-Party Agreement requirement-driven TWRS Characterization Program information.

Brown, T. M., J. W. Hunt, and L. J. Fergestrom, 1997, *Tank Characterization Technical Sampling Basis*, HNF-SD-WM-TA-164, Rev. 3, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Summarizes the 1997 technical basis for characterizing tank waste and assigns a priority number to each tank.

Brown, T. M., J. W. Hunt, and L. J. Fergestrom, 1998, *Tank Characterization Technical Sampling Basis*, HNF-SD-WM-TA-164, Rev. 4, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Summarizes the 1998 technical basis for characterizing tank waste and assigns a priority number to each tank.

Buckley, L. L., 1997, *Vapor Sampling and Analysis Plan*, HNF-SD-WM-TSAP-126, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains vapor sampling and analysis procedure for 200 Area Tanks.

Crawford, B. A., 1998, *Tank 241-AX-104 Residual Solids Leach Tests*, HNF-SD-HTI-TP-001, Rev. 1, Numatec Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Describes the test plan for the leach test on the composited waste from the 1997 auger sampling event.

DOE-RL, 1996, *Recommendation 93-5 Implementation Plan*, DOE/RL-94-0001, Rev. 1, U.S. Department of Energy, Richland, Washington.

- Describes the organic solvents issue and other tank issues.

Hall, K. M., 1998, *Extension of Tank 241-AX-104 Format III Report Deadline*, (internal memorandum 7A120-98-003 to R. A. Esch, K. L. Powell, and C. M. Seidel, January 19), Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Extended the Format III report deadline because of delays in sample homogenization and subsampling of the 1997 auger samples.

Schreiber, R. D., 1998, *Tank 241-AX-104 Auger Sampling and Analysis Plan*, HNF-SD-WM-TSAP-149, Rev. 0A, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains sampling and analysis requirements for tank 241-AX-104 based on applicable DQOs.

Schreiber, R. D., 1998, *Tank 241-AX-104 Light Duty Utility Arm Sampling and Analysis Plan*, HNF-2071, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains requirements for collecting and analyzing samples from tank 241-AX-104 obtained using the light duty utility arm.

Schreiber, R. D., 1998, *Revision to Tank 241-AX-104 Analytical Requirements Because of Sample Handling Difficulties*, (internal memorandum 7A110-98-005 to R. A. Esch and D. B. Hardy, March 18), Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains changes made to the analytical plan for the 1997 auger samples because of sample handling difficulties.

Schreiber, R. D., 1998, "Revision to Tank 241-AX-104 Leach Test Requirements," (internal memorandum 7A110-98-014 to B. A. Crawford and R. A. Esch, May 21), Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains changes made to the leach test analytical plan for the 1997 auger samples because of representativeness concerns for the riser 3A samples.

**Ie. Data Quality Objectives and Customers of Characterization Data**

Banning, D. L., 1998, *Tank 241-AX-104 Waste Characterization Data Quality Objective*, HNF-SD-WM-DQO-027, Rev. 0B, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Defines the amount, type, and quality of information needed to characterize the residual waste in tank 241-AX-104 in support of the Hanford Tanks Initiative Project.

Banning, D. L., 1998, *Hanford Tank Initiative Tank 241-AX-104 Upper Vadose Zone Demonstration Data Quality Objectives*, HNF-2326, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Evaluates the use of cone penetrometer technologies and provides information supporting vadose zone soils characterization adjacent to tank 241-AX-104.

Dukelow, G. T., J. W. Hunt, H. Babad, and J. E. Meacham, 1995, *Tank Safety Screening Data Quality Objective*, WHC-SD-WM-SP-004, Rev. 2, Westinghouse Hanford Company, Richland, Washington.

- Determines whether tanks are under safe operating conditions.

Meacham, J. E., D. L. Banning, M. R. Allen, and L. D. Muhlestein, 1997, *Data Quality Objective to Support Resolution of the Organic Solvent Safety Issue*, HNF-SD-WM-DQO-026, Rev. 0, DE&S Hanford, Inc. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains requirements for the organic solvents DQO.

Osborne, J. W., and L. L. Buckley, 1995, *Data Quality Objectives for Tank Hazardous Vapor Safety Screening*, WHC-SD-WM-DQO-002, Rev. 2, Westinghouse Hanford Company, Richland, Washington.

- Contains requirements for addressing hazardous vapor issues.



Schreiber, R. D., 1997, *Memorandum of Understanding for the Organic Complexant Safety Issue Data Requirements*, HNF-SD-WM-RD-060, Rev. 0, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains requirements, methodology and logic for analyses to support organic complexant issue resolution.

## II. ANALYTICAL DATA - SAMPLING OF TANK WASTE AND WASTE TYPES

### IIa. Sampling of Tank 241-AX-104

Buckingham, J. S., 1978, *Heat Generation of Residual Sludge in Tank 104 AX*, (letter 60120-78-040 J to C. D. Campbell, June 15), Rockwell Hanford Operations, Richland, Washington.

- Presents a heat generation estimate based on a sample of the residual tank sludge; however, the specific sampling event is unknown. Also presents the results of an analysis of sludge in tank 004-AR, which contained sluiced 241-AX-104 material. A heat generation rate estimate was derived based on this analytical data.

Crawford, B. A., 1998, *Tank 241-AX-104 Residual Solids Leach Test Results*, TWR-3548, Rev. 0, Numatec Hanford Corporation for Fluor Daniel Hanford, Inc., Richland, Washington.

- Presents results from the composite and leach test analyses on the November 1997 auger samples.

Esch, R. A., 1998, *Tank 241-AX-104, Auger Samples, 97-AUG-001, 97-AUG-002, 97-AUG-003, and 97-AUG-004 Analytical Results for the Final Report*, HNF-SD-WM-DP-298, Rev. 0, Waste Management Federal Services of Hanford Inc. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains results for the analysis of the individual 1997 auger samples.

Horton, J. E., and J. S. Buckingham, 1974, *Characterization and Analysis of Tank 104-AX Sludge*, (letter to O. R. H. Rasmussen, October 14), Atlantic Richfield Hanford Company, Richland, Washington.

- Presents analytical results for a May 1974 sludge sample.

Koegler, S. S., 1976, *A and AX Tank Sludge Heat Generation Rates*, (letter to R. E. Felt, February 12), Atlantic Richfield Hanford Company, Richland, Washington.

- Calculates a heat generation rate based on the  $^{90}\text{Sr}$  and  $^{137}\text{Cs}$  data from the May 1974 sludge sample.

Lockrem, L. L., 1997, *Revised Data Tables for Tank Vapor Database on Tanks 241-A-106, 241-AX-104, and 241-TX-106*, (letter NHC-9756182 to M. R. Adams, July 17), Numatec Hanford Corporation for Fluor Daniel Hanford, Inc., Richland, Washington.

- Provides revised data tables from the January 1997 vapor sampling event.

Starr, J. L., 1977, *104-AX Sludge Analysis*, (letter to F. M. Jungfleisch, October 14), Rockwell Hanford Operations, Richland, Washington.

- Presents analytical results for a September 1977 sludge sample.

Viswanath, R. S., G. S. Caprio, J. G. Douglas, M. J. Duchsherer, E. S. Mast, L. A. Pingel, M. Stauffer, D. B. Bonfoey, and G. A. Fries, 1998, *Tank Vapor Sampling and Analysis Data Package for Tank 241-AX-104, Sampled January 23, 1997*, HNF-SD-WM-DP-278, Rev. 0, Numatec Hanford Corporation for Fluor Daniel Hanford, Inc., Richland, Washington.

- Presents field data and analytical results from the January 23, 1997 headspace vapor sampling of tank 241-AX-104.

Wheeler, R. E., 1975, *Analysis of Tank Farm Samples*, (letter to R. L. Walser, October 2), Atlantic Richfield Hanford Company, Richland, Washington.

- Contains results from a 1975 liquid sample.

## **IIb. Sampling of PUREX High-Level Waste**

Buckingham, J. S., 1978, *Heat Generation of Residual Sludge in Tank 104 AX*, (letter 60120-78-040 J to C. D. Campbell, June 15), Rockwell Hanford Operations, Richland, Washington.

- Presents a heat generation estimate based on a sample of the residual tank sludge; however, the specific sampling event is unknown. Also presents the results of an analysis of sludge in tank 004-AR, which contained sluiced 241-AX-104 material. A heat generation rate estimate was derived based on this analytical data.

Van Tuyl, H. H., 1958, *Composition of Some PUREX Plant IWW Solutions*, HW-57280, General Electric Company, Richland, Washington.

- Presents compositions of some of the PUREX Plant IWW (now known as P2) solutions.

## **III. COMBINED ANALYTICAL/NON-ANALYTICAL DATA**

### **IIIa. Inventories from Campaign and Analytical Information**

Agnew, S. F., J. Boyer, R. A. Corbin, T. B. Duran, J. R. Fitzpatrick, K. A. Jurgensen, T. P. Ortiz, and B. L. Young, 1997, *Hanford Tank Chemical and Radionuclide Inventories: HDW Model Rev. 4*, LA-UR-96-3860, Rev. 0, Los Alamos National Laboratory, Los Alamos, New Mexico.

- Contains waste type summaries and primary chemical compound/analyte and radionuclide estimates for sludge, supernatant, and solids.

Allen, G. K., 1976, *Estimated Inventory of Chemicals Added to Underground Waste Tanks, 1944 - 1975*, ARH-CD-601B, Atlantic Richfield Hanford Company, Richland, Washington.

- Contains major components for waste types, and some assumptions. Purchase records are used to estimate chemical inventories.

Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1996, *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area*, WHC-SD-MW-ER-349, Rev. 0A, Westinghouse Hanford Company, Richland, Washington.

- Contains summary information from the supporting document as well as in-tank photo collages and the solid composite inventory estimates Rev. 0 and Rev. 0A.

### **IIIb. Compendium of Data from Other Physical and Chemical Sources**

Brevick, C. H., J. L. Stroup, and J. W. Funk, 1997, *Supporting Document for the Historical Tank Content Estimate for AX-Tank Farm*, WHC-SD-WM-ER-309, Rev. 1B, Fluor Daniel Northwest for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains historical data and solid inventory estimates. The appendices contain level history AutoCAD sketches, temperature graphs, surface level graphs, cascade/dry well charts, riser configuration drawings and tables, in-tank photos, and tank layer model bar charts and spreadsheets.

Brevick, C. H., L. A. Gaddis, and E. D. Johnson, 1995, *Tank Waste Source Term Inventory Validation, Vol I & II*, WHC-SD-WM-ER-400, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

- Contains a quick reference to sampling information in spreadsheet or graphical form for 23 chemicals and 11 radionuclides for all the tanks.

Hanlon, B. M., 1997, *Waste Tank Summary Report for Month Ending September 30, 1997*, WHC-EP-0182-126, Lockheed Martin Hanford Corp. for Fluor Daniel Hanford, Inc., Richland, Washington.

- Contains a monthly summary of the following: fill volumes, Watch List tanks, occurrences, integrity information, equipment readings, equipment status, tank location, and other miscellaneous tank information.

Husa, E. I., 1993, *Hanford Site Waste Storage Tank Information Notebook*, WHC-EP-0625, Westinghouse Hanford Company, Richland, Washington.

- Contains in-tank photographs and summaries on the tank description, leak detection system, and tank status.

Husa, E. I., 1995, *Hanford Waste Tank Preliminary Dryness Evaluation*, WHC-SD-WM-TI-703, Rev. 0, Westinghouse Hanford Company, Richland, Washington.

- Assesses relative dryness between tanks.

LMHC, 1998, *Tank Characterization Data Base*, Internet at <http://twins.pnl.gov:8001/htbin/TCD/main.html>

- Contains analytical data for each of the 177 Hanford Site waste tanks.

Shelton, L. W., 1996, *Chemical and Radionuclide Inventory for Single- and Double-Shell Tanks*, (internal memorandum 74A20-96-30 to D. J. Washenfelter, February 28), Westinghouse Hanford Company, Richland, Washington.

- Contains a tank inventory estimate based on analytical information.

Van Vleet, R. J., 1993, *Radionuclide and Chemical Inventories*, WHC-SD-WM-TI-565, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

- Contains tank inventory information.

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